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# Japan Report

## SCIENCE AND TECHNOLOGY





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# JAPAN REPORT

## SCIENCE AND TECHNOLOGY

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# ARTIFICIAL MEMBRANE AT CELL LEVEL DISCUSSED

Tokyo BIO INDUSTRY in Japanese Jul 86 pp 31-36

[Text] Studies on artificial membranes mimicking biomembrane functions have been conducted recently. Particularly, the separation of materials by artificial membranes with carriers which imitate the very high, specific biomembrane transport system is expected to be used for practical applications, and has been attracting much attention. In this report, the future prospects of impregnate liquid membranes, which will be used for optical separation of amino acids, are discussed.

## 1. Biomembrane and Artificial Membrane

The biomembrane, an element of cell structure, not only partitions a cell, but it also has important functions concerning life itself. These functions are the transport of particular ions and materials, the recognition and exchange of information, the production of biological substances, energy conversion, and the membrane movement induced by the cytoskeleton. Basically, the biomembrane consists of two molecular layers of polar lipids; its functions, however, are carried out by various proteins in the membrane.

What is required to imitate and apply the biomembrane are to design the compounds and the chemical reactions substituting the functions of the proteins in the membrane, and to prepare the site to express the functions. Based on these views, various studies on the artificial membrane and surrounding technologies have been carried out: for instance, separating membranes, sensors, membrane reactors, catalysts moving between phases, and energy conversion. Because of the limited length of the report, only the separating membranes are discussed here. There is a lot of excellent, recommended literature relating to the other technologies.

The characteristics of transport by the biomembrane which the separating membrane will imitate are:

(1) high selectivity;
(2) active transport and facilitated transport linked to the consumption of various types of energy, and
(3) asymmetry of the biomembrane structure and fact that some transport systems have a definite direction for transport.

These characteristics originate from the properties of proteins in the membrane, or transporters. The primary structures of some transporters, such as Na channel and lactate transporter, were identified by gene recombination techniques, but their higher structures and functions are not yet known. On the other hand, the relationship between structures and functions is well known for ionophores with low molecular weights, such as a carrier like valinomycin, and a channel-forming molecule like gramicidin, which have been studied as carriers mediating transport in biomembrane models. Starting from these studies, various separating membranes with carriers have been devised so far.

## 2. Separating Membranes With Carriers

Separating membranes with carriers are divided into two groups: one having fixed carriers and the other with carriers which diffuse in the membrane.

Most of the separating membranes with fixed carriers are made of high molecular membranes. In one method, carriers are fixed on high molecular chains by chemical bonding; in the other, carriers are blended when the membrane is produced. The advantages of this type are that the carriers do not elude and that the membranes are very strong. In addition, this kind of membrane is easily changed to asymmetric forms, but this characteristic is used only in a few cases. These membranes, however, always have technical problems, for instance, relating to membrane function as the permeate-barrier which is important to advance selectivity, and the way to arrange several carriers.

The separating membranes with moving carriers are generally liquid membranes. The liquid membranes include bulk liquid membrane like chloroform, impregnate liquid membrane, high-molecular plastic membrane, liquid crystal membrane, w/o/w emulsion, and ribosomes. Since liquid membranes are easily formed, the focus of interest lies in the design and synthesis of carriers rather than the development of materials for membranes. The problems of liquid membranes are discussed later in this report.

In both fixed and moving carrier types, the choice of a carrier depends on materials to separate. The one thing we expect is the extremely selective permeability for isomers. The majority of cases of the carrier mediated transport of organic compounds are found in liquid membranes; they are the transport of optically separated amino acids by photoactive crown ether (discussed later), the transport of mandelic acid by photoactive amines, ADP transport by diamine, and the transport of sugars by phenylboric acid. These are performed with bulk liquid membranes in order to test the functions of carriers. Recently, practical use of liquid membranes has been tried and impregnate liquid membranes have attracted attention.

## 3. Impregnate Liquid Membranes

The impregnate liquid membrane is the liquid membrane formed in the pores of a multiporous supporter. The membrane is used as the model for the biomembrane in basic research, while it is also studied with the view of practical chemical engineering applications. Particularly, studies on the recovery and

condensation of metal ions with chelating agents used as carriers have been conducted recently.

The comparison between the impregnate liquid membrane and the biomembrane shows that the liquid membrane substitutes the functions of each of the constituents of the biomembrane (Figure 1 shows the impregnate liquid membrane used for the transport of amino acids which are separated optically). The aspects of functional substitution are not very different in other liquid membranes.

However, different from the biomembrane and the artificial membranes with fixed carriers, it is generally difficult to change the forms of liquid membranes to asymmetrical ones. Therefore, the aqueous phases of both sides of a membrane are usually made to have different constituents in order to conduct active transport (in broader sense) through liquid membranes. The differences in ionic level are used as the driving force for neutralizing and oxidation-reduction reactions. On the otherhand, there is another method in which the affinity between the carrier and the substance to be transported is changed by using optical energy and temperature gradients; in this case, the constituent of both aqueous phases are allowed to have the same constituents.

Impregnate liquid membrane

Biomembrane

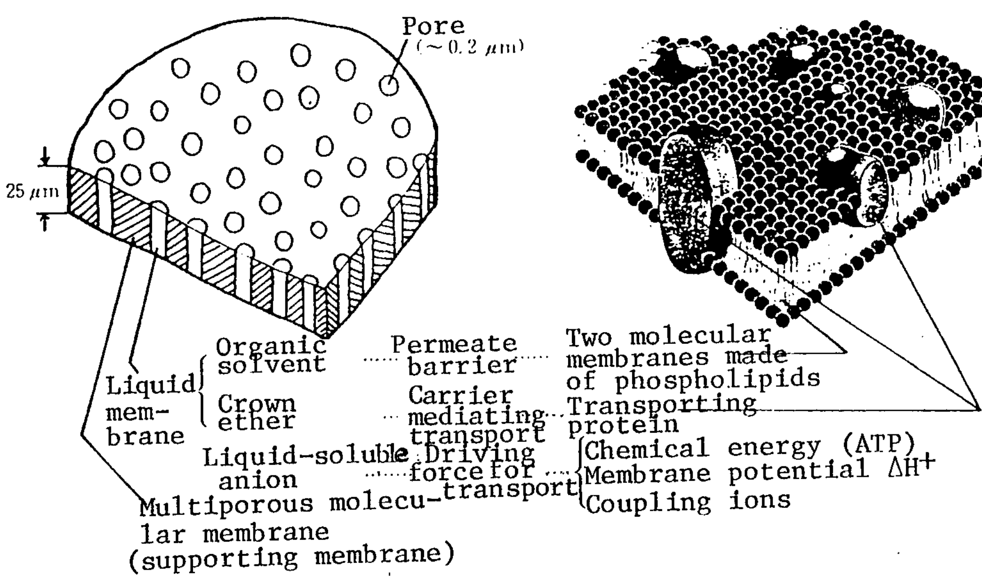


Figure 1. Comparison Between Biomembrane and Impregnate Liquid Membrane

In addition to the problems common to liquid membranes, the particular problems for impregnate ones are the stability of liquid membranes and the elusion of carriers. It is caused by the extremely large volume ratio between the aqueous phase and the liquid membrane compared with the bulk liquid membrane. For this reason, it will be required to study the affinity between the multiporous film as a supporter and the solvent of the liquid membrane, and to prevent the elusion of carriers.

## 4. The Membrane to Separate Optical Isomers of Amino Acids

Now let us consider, by showing some practical examples, the future prospects and some problems of artificial membranes developed by the imitation functions of the biomembrane. In the section below, the membranes used to separate optical isomers of amino acids, which the present authors are studying, will be introduced.

### 4.1 Amino Acid Transport and Photoactive Crown Ether

Following the time process of how the transport of optically separated amino acids has been developed is very interesting, and therefore it is briefly referred to in this section.

It is Moore and Schechter who succeeded in active transport through artificial membrane which substitutes the functions of the biomembrane. Four years later, in 1973, Behr and Lehn reported the transport of amino acids. It was a system where the anions of dinonylnaphthalene sulfonic acid or the cations of trioctyl-methyl-ammonium are dissolved as carriers in the bulk liquid membrane of toluene. In 1974, 7 years after the discovery of crown ether by Pedersen, Cram reported the transport of optically separated phenyl-glycine-methyl-ester through the bulk liquid membrane with photoactive crown ether [1] used as a carrier. The crown ether which Cram used was very well designed (the principle of optical separation by photoactive crown ether has been described in detail in separate literature). They obtained a value of 11.5 as the ratio of optical separation of racemic compounds, which has never been obtained since then. Probably for this reason, the transport of amino acids has been studied little.

In 1981, Cram reported the synthesis of crown ether [2]. The extraction showed that it had very high power to distinguish optical isomers, though it had only one asymmetric carbon atom in it. Since 1983, Nakazaki, Yamamoto, and Inamura of Osaka University have synthesized several kinds of active crown ether. Among them, compound [3] has approximately the same ratio of transportation as compound [1].

While, in 1971, Cussler reported the transport of Na with (monensin), in which he referred to the purpose of his study as having been optical separation of lisine through a high molecular supporting membrane. Ten years later, the transport of amino acids by a high molecular plastic membrane was reported by Sugiura. This membrane system consists of phosphoric trialkyl ester and PVC. This system was also applied to the impregnate liquid membrane, and its use as the liquid membrane solvent was also studied. Thus, Cussler's aim has become practical.

### 4.2 Optical Separation of Amino Acids by Impregnate Liquid Membrane

#### (1) Cotransport System

The impregnate liquid membrane used for the transport of optically separated amino acids is prepared by impregnating O-nitro-phenyl-ether dissolved as a

carrier into a polypropylene multiporous film (Duraguard #2400). The crown ether suitable for this system, is shown in (2) of Figure 2. (1) combines scarcely with amino acids, and therefore it is not suitable as a carrier. (3) is also a good one, but (2) has a larger separation ratio. Table 1 shows the results of the transport of optically separated amino acids combined with $ClO_4^-$ by using the impregnate liquid membrane in which 50 mM of crown ether (2) is dissolved.

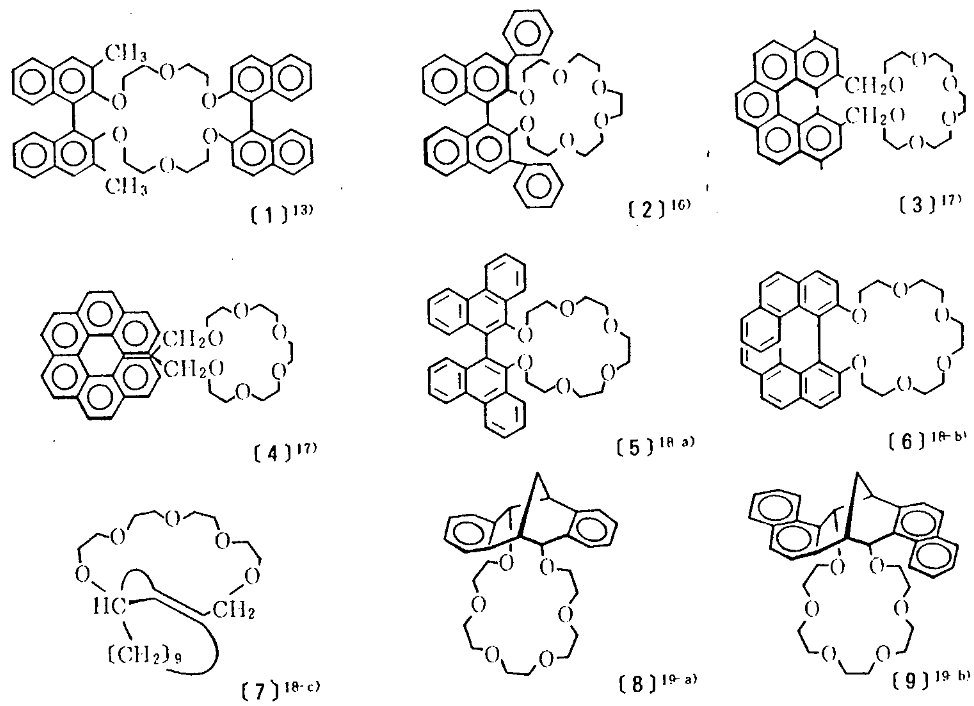


Figure 2. Photoactive Crown Ether

In this impregnate liquid membrane, an amino acid, not its ester, can be separated and transported under acidic conditions. As seen in the case of phenylglicine, the membrane also carries out optical separation with the optical purity of 91.6 percent in one operation, which is attributable to the excellent properties of crown ether (2).

The flux ratio between D-compound and L-compound when the racemic body is used for measurement (the ratio of optical separation) and that when D- or L-compound is used individually for measurement are not the same (marked rac. or D/L as experimental condition); the racemic body gives a larger value. It suggests that the competitive transport mechanism, which sometimes occurs in the biomembrane, is also seen in this liquid membrane. The value of the optical separation ratio for the racemic body is equal to the value of the distribution ratio of optical isomers which Cram showed with extraction experiments. It is natural to consider that the distribution equilibrium of optical isomers on the membrane surface is set, and the distribution ratio determines the ratio of optical separation. In another experiment,

Table 1. Optical Separation of Racemic Amino Acids by Impregnate Liquid Membrane in Which (2) in Figure 2 is Dissolved 50mM

| Amino acids | Experimental condition | Time passed (expressed in hour) | Flux ($10^{-7}$ mol cm$^{-2}$h$^{-1}$) | | |
|---|---|---|---|---|---|
| | | | $J_D$ | $J_L$ | $J_D/J_L$ |
| Phenylglycine | rac | 12.7 | 5.97 | 0.26 | 22.7 |
| | D/L | 12.7 | 6.28 | 1.34 | 4.7 |
| Tryptophan | rac | 12.7 | 4.66 | 0.69 | 6.7 |
| | D/L | 12.7 | 4.68 | 1.25 | 3.7 |
| Phenylalanine | rac | 12.7 | 3.99 | 0.55 | 7.2 |
| | D/L | 12.7 | 4.80 | 1.42 | 3.4 |
| Leucine | rac | 25.5 | 3.87 | 0.29 | 13.5 |
| | D/L | 12.0 | 4.34 | 0.97 | 4.5 |
| iso-Leucine | rac | 12.0 | 1.11 | 0.07 | 15.0 |
| tert-Leucine | rac | 25.5 | 0.42 | 0.03 | 13.4 |
| | D/L | 24.0 | 0.44 | 0.06 | 6.9 |
| Methionine | rac | 25.3 | 3.40 | 0.32 | 11.2 |
| | D/L | 12.0 | 3.77 | 0.85 | 4.4 |
| Valine | rac | 25.5 | 0.75 | 0.10 | 7.7 |
| | D/L | 24.0 | 0.78 | 0.14 | 5.8 |
| Tyrosine | rac | 47.0 | 0.26 | 0.04 | 6.0 |

Experimental conditions···rac: aqueous phase I = $0.1_M$ racemic amino acid/$0.05_M$ $HClO_4$, D/L: aqueous phase I = $0.05_M$ D- or L-amino acid/$0.05_M$ $HClO_4$, both aqueous phase I and II contain $0.05_H$ $H_2SO_4$. 25°C

it is found that the molecular diffusion in the membrane is a rate-determining factor of transport. This supports the view mentioned previously.

The rise in the concentration of $ClO_4^-$ compared with racemic compounds enables the transport by optical separation along with active transport (Figure 3). At a certain point (after 14 hours in the figure), one of the isomers is transported against the concentration gradient, and finally D- and L- phenylglycine, with the optical purity of 76 percent and 58 percent, respectively, being simultaneously obtained.

(2) Countercurrent Transport System

In a cotransport system, amino acids are transported with lipid-soluble anions. Therefore, optically separated amino acids are obtained as salts of lipid-soluble anions.

As a transport system in which electrical neutrality is maintained, there is countercurrent transport other than cotransport. In this system, ions or electrons having the same electrical charge are transported in the opposite direction, which is often seen in the biomembrane. In order to form a

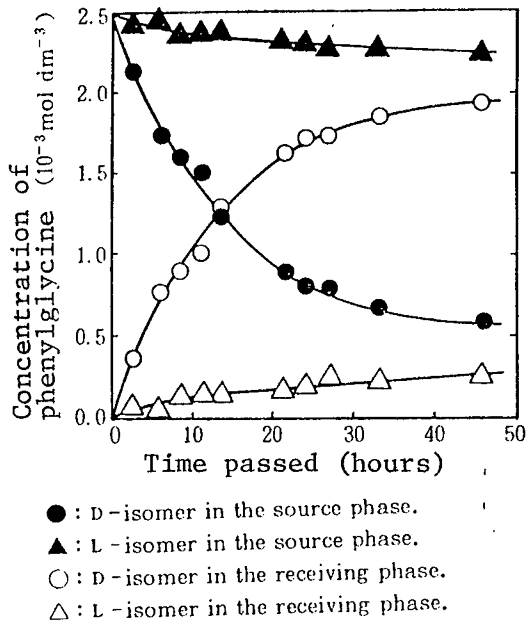


Figure 3. Transport of Phenylglycine by Optical Separation Coupling With Active Transport

countercurrent transport system in an artificial membrane, the ions oppositely charged (e.g., anions) from the ions to be transported (e.g., cations) are required to be present as carriers in the membrane.

It is found that when the anions of dinonylnaphthalene sulfonic acid are added to the previously discussed impregnate liquid membrane, amino acids are optically separated and transported against the concentration gradient of alkaline metal ions and hydrogen ions. It is also found that the kind of anions in the amino acid receiving phase does not affect the transport system, which is very convenient for practical use.

## 5. Task and Prospects in the Future

Membrane-separation is suitable for large-scale treatment, and it is also used for repetitive treatment. It is expected that when the separation ratio for one membrane-separation treatment is $\alpha$, the ratio will rise to $\alpha^n$ by the treatment repeated n times. From this viewpoint, it is very provable that amino acids can be optically separated by using membranes.

However, there are a few problems yet to be solved, such as the rise in liquid membrane stability, and the way of preventing the elusion of carriers. If the elusion of carriers to the aqueous phase is extremely small, the reimpregnation of only the liquid membrane solvent can recover the membrane functions; this must be fully studied along with its application form. The rate of transport is not satisfactory, and therefore, the effort to widen the effective area of a membrane as well as the study of ways of making it thin will be required.

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## FRICTION WITH UNITED STATES OVER SEMICONDUCTORS DISCUSSED

Tokyo DENSHI KOGYO GEPPO in Japanese No 4, 1986, pp 2-9

[Article by Director Yukio Shimura of Kogyō Chosa Kai Co., Ltd.: "Background and Future Problems of U.S.-Japan Semiconductor Trade Friction"]

[Excerpts] We can see a certain regularity in the process by which trade friction between the United States and Japan was generated. In the 1960s the raw material industry became a target and friction in connection with textiles, iron and steel developed. In the 1970s, the focus of the friction moved to the assembly processing-type industry, especially durable consumer goods, and color televisions and motor vehicles became involved. Friction in the 1980s involves high-tech industry; friction in connection with semiconductors, communications, numerical control (NC), machine tool, etc., has already arisen. Because the semiconductor is a key high technology, the "rice of industry," the tension is extreme.

In this article, I would like to describe the background of the semiconductor issue between the United States and Japan and shed some light on present and future problems.

### U.S. 'High-Tech' Industry--Sense of Mounting Crisis

This writer visited the United States in late November last year as a leader of the delegation of the Microelectronics Study Association to survey the United States under the sponsorship of the Software Application Economy Center. We surveyed the development of U.S. microelectronics and the broader situation of the U.S. business world. It appeared that the U.S. electronics industry had lost its vitality, adversely affected by the "unprecedented recession" (Chairman G. Moore of Intel Corp.) and had become an "ailing giant."

This was strongly felt in the keynote address by President J. Young of Hewlett-Package Co. at the "Autumn Comdecs '85." In his talk, "Revival From Industrial Stagnation," he emphasized the sense of crisis that "the United States last year (1984) recorded $15 billion deficit in terms of electronics trade with Japan. This monetary value is larger than the deficit generated by motor vehicle trade worldwide. Silicon Valley is now going to become another Detroit" (see Figure 1).

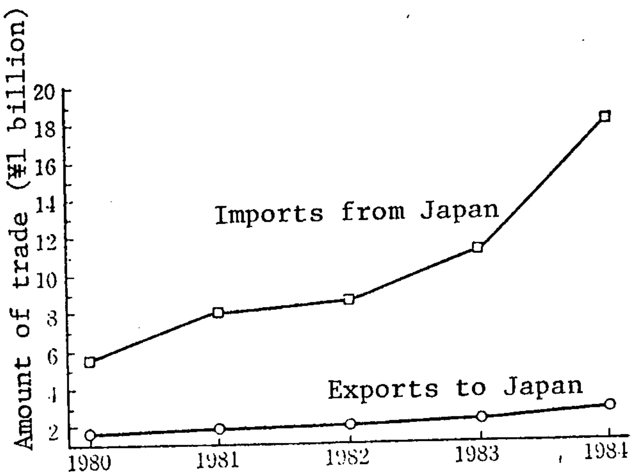


Figure 1. U.S. Electronics Trade Imports and Exports With Japan

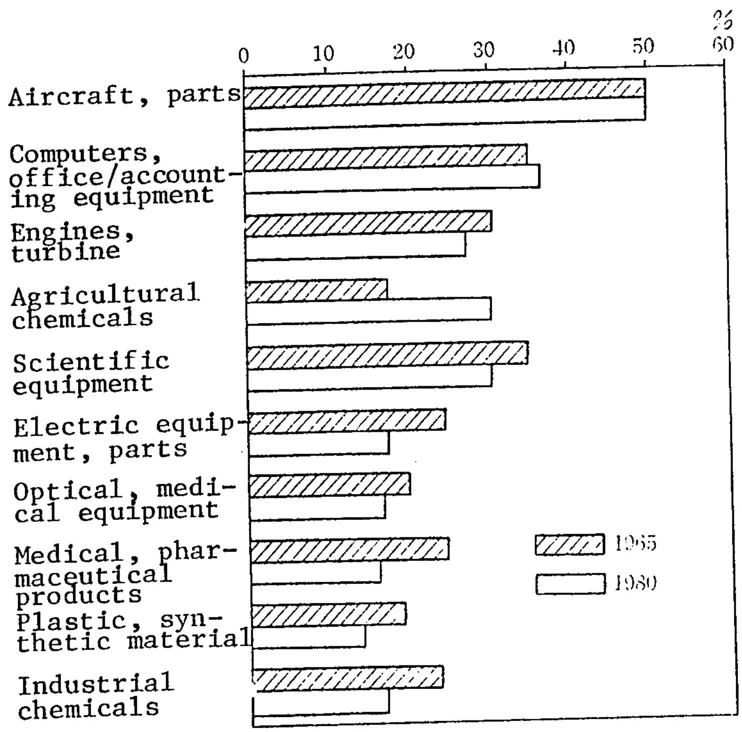


Figure2. U.S. World Export Shares of High-Tech Products (Comparison between 1965 and 1980)

Figure 2 shows the U.S.'s share of high-tech exports in 1965 and 1980. The figure gives 10 branches of the high-tech field said to be the "sunrise (growth) branches"; only three branches, aircraft, computers, and agricultural use chemicals, remained the same or increased world market share throughout the period.

Of grave concern is the fact that such a lowering of the position in the market has been caused, by the relative decline in the technological strength. This is indicated in the report entitled "Evaluation of Competitive Strength of the United States in High-Tech Industry" worked out by the U.S. Commerce Department in February 1983. In the report, the nine high-tech industries were taken up and comparisons with Japan and Europe (particularly Japan) were made. In particular, with regard to computers, although admitting that the United States is in a relatively superior position, "the United States is maintaining its wideranging superiority," a sense of crisis is openly expressed over the fact that Japan is rapidly catching up, "Japan is displaying its strength in point of applying high performance to computer's mainframe"; "Each Japanese maker has a product that can compete with that of major U.S. makers"; and "In terms of the supercomputer also, a Japanese maker has a model that can compete with that of the United States."

With regard to optical communications, too, the report recognizes, as expected, that the Japanese group is fighting a good fight, "Japan stands superior in terms of optical source technology and its application; with regard to other technologies (transmission, etc.), too, Japan is competing with the United States." Also, with regard to semiconductors, while admitting that "the United States stands superior in terms of microprocessor technology," the report says definitely that "the United States already lost its superiority in several fields of semiconductor technology" and highly evaluates Japan's rise in position especially in fields such as metal-oxide semiconductor (MOS) very large-scale integrated circuit (LSI) memory, complementary metal-oxide semiconductor (CMOS) technology, and semiconductor manufacturing equipment. In the summary, the report warned that "comparative superiority (of the United States in the high-tech field) has already begun to collapse" and concluded that the "challenge of major technology to the United States comes from Japan. At present, the challenge is limited to a small number of advanced technological fields, but in the future, this challenge is expected to expand to more extensive fields."

It seems that the United States has now begun to realize that it is on the verge of a stagnation in the high-tech area in which it should maintain its absolute superiority.

Domestic Semiconductors Threatening the United States

Japan's position in the semiconductor trade problem may be compared to that of a child whose parent, the United States, has brought it up, but the child is now as big as the parent and may occasionally challenge him. If the position of the chaser improves, the sense of crisis in the one being chased will be heightened. The semiconductor friction between the United States and Japan is, frankly, inevitable.

The situation inwhich Japan is changing from relative inferiority to relative superiority can be corroborated by several figures.

One thing is that the percentages of production between the United States and Japan are coming closer. According to Dataquest Inc. of the United States

the ratio of amount of shipment by maker affiliation in 1985 (total amount of shipment is $24 billion) included 48 percent of the U.S.-affiliated makers and 42 percent of the Japanese-affiliated makers; the difference between the two was six points. With regard to 1980 (total amount of shipment is $13.9 billion), the U.S. side was 61 percent and the Japanese side 26 percent, and the difference was 35 points. Even for 1984 (total amount of shipment is $28.8 billion), the U.S. side was 51 percent and the Japanese side was 39 percent, a 12-point difference. Therefore, it can be seen that Japan is rapidly catching up with the United States in recent years (see Figure 3). In spite of that, the United States still ranks above Japan; if the United States is regarded as yokozuna (grand champion of sumo), Japan may be regarded as ozeki (champion of sumo). The European group that occupies less than 10 percent may be seen as corresponding to No 10 maegashira (senior grade sumo) at best.

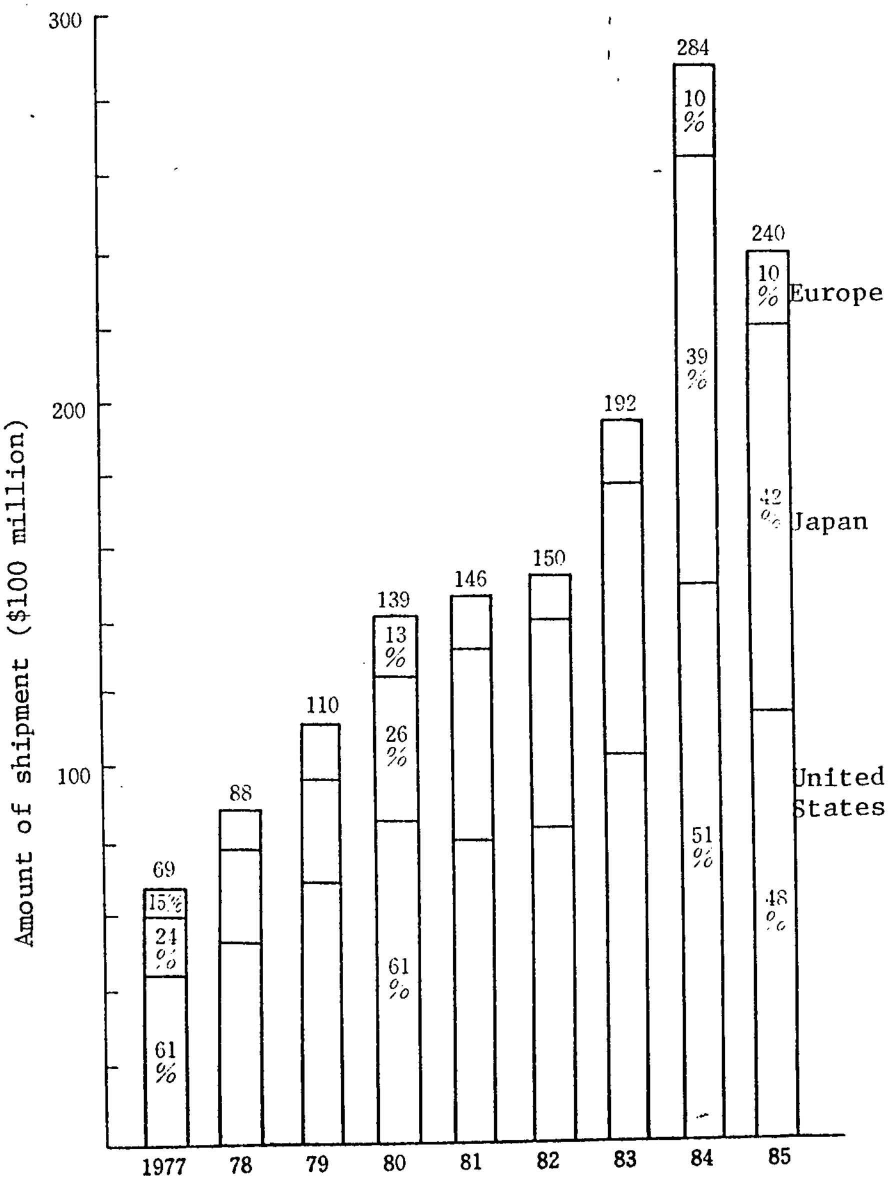


Figure 3. Trend of World Semiconductor Amount of Shipment by Affiliation of Makers (U.S. Dataquest Inc.)

The problem is the fact that Japan in the rank of ozeki has begun to display its ability to overwhelm even yokozuna sometimes.

What showed this distinctly is the sales rankings by makers for 1985 worked out by Dataquest Inc. (see Table 1). U.S. Texas Instruments (TI) that had defended its top-ranked position since the transistor era slipped; in its stead was NEC, a top domestic maker. TI, ranking first last year, decreased 28.8 percent from the previous year and dropped to third place. NEC, ranking third last year, held the decrease to 11.9 percent, indicating the real situation of the change in the top rank. With regard to other Japanese firms, Hitachi and Toshiba remain in the same rankings as the previous year; Fujitsu advanced from 9th place to 7th and the Matsushita group from 12th to 10th. A total of 5 firms are listed in the best 10. In addition, in 11th place and higher, Mitsubishi Electric Corp. ranks 11th, the Sanyo group 14th, Sharp Corp. 16th, and Oki Electric Industry 19th. The so-called 9 major firms are all listed from 20th place and above. On the contrary, with regard to the U.S. firms, excluding Motorola ranking 2d and Intel ranking 8th, National Semiconductor lowered its ranking to 9th from 7th from the previous year and AMD to 12th from 10th; so the degree of these declines is remarkable.

Table 1. Trend of Semiconductor Sales of World Major Makers

(Surveyed by Dataquest Inc.)
(Unit: $1 million; percent)

| Ranking in 1985 | Name of firm | Sales in 1985 | Increase/decrease compared with previous year |
|---|---|---|---|
| 1 (3) | NEC Corp. | 1,984 | ▲11.9 |
| 2 (2) | Motorola Inc. | 1,850 | ▲20.3 |
| 3 (1) | Texas Instruments, Inc. | 1,766 | ▲28.8 |
| 4 (4) | Hitachi, Ltd. | 1,671 | ▲18.6 |
| 5 (5) | Toshiba Corp. | 1,459 | ▲ 6.5 |
| 6 (6) | (Philips Signetics) | 1,068 | ▲19.4 |
| 7 (9) | Fujitsu, Ltd. | 1,020 | ▲14.3 |
| 8 (8) | Intel Corp. | 1,020 | ▲15.1 |
| 9 (7) | National Semiconductor Corp. | 940 | ▲25.6 |
| 10 (12) | Matsushita Electric Industrial Co., Ltd./Matsushita Electronics Corp. | 906 | ▲ 2.4 |

▲ indicates decrease; figures in parentheses indicate rankings in 1984

There are two reasons why Japanese makers fought a good fight in the midst of the so-called unprecedented semiconductor recession.

One reason is that under the same semiconductor recession, whereas the industry-oriented U.S. market fell into a destructive situation suffering a direct hit from "computer slumps" (BUSINESS WEEK), the Japanese market was supported by the two markets of industrial and consumer use that play the role of "two wheels of a vehicle"; the injury the Japanese market suffered from the recession was comparatively slight. In fact, with regard to Japan's electronics

industry production in 1985, electronic parts ended in minus growth, 1.4 percent decrease, compared with the previous year, but electronic equipment for consumer use increased by 4.6 percent and electronic equipment for industrial use increased by 13.3 percent, producing fair results.

The second reason Japanese makers have fought the good fight is the fact that Japanese makers are displaying their abilities more and more in the DRAM market and is shutting out the European and U.S. firms. Above all, with regard to the 256K-bit products whose full-scale mass production began last year, Japanese makers this year produced 630 million units of 690 million units, the total amount of shipment throughout the world, which accounts for 91 percent share; thus they are expected to almost monopolize the market. The only firm that is putting up a good fight in the U.S. group is TI, but its production stronghold is TI, Japan.

Not only that, with regard to the 1M-bit products, strategical products following the 256K-bit products, major domestic firms beginning with Toshiba have begun sample shipment ahead of other manufacturers and will cover the entire amount of shipment of 3 million units (see Table 2, surveyed by Nikkei Telecom). It may be said that domestic producers in the semiconductor business have secured superiority in the "memory" market.

Table 2. Estimates of Amount of DRAM Shipment in World Market (1986)

(Unit: 1 million)

| | World | Japan | United States | Europe | Southeast Asia |
|---|---|---|---|---|---|
| 4K | 3 (15) | 1 (3) | 1 (10) | 1 (2) | 0 (0) |
| 16K | 25 (75) | 10 (10) | 10 (60) | 5 (5) | 0 (0) |
| 64K | 250 (895) | 100 (435) | 50 (400) | 50 (30) | 50 (30) |
| 256K | 690 (381) | 630 (326) | 40 (50) | 0 (0) | 20 (5) |
| 1M | 3 (0) | 3 (0) | 0 (0) | 0 (0) | 0 (0) |
| Total | 971 (1,366) | 744 (774) | 101 (520) | 56 (37) | 70 (35) |

Figures in parentheses are actual results in 1985.
Note: The amount of shipment in the outside-sale market excluding in-house manufacturers.
Surveyed by Nikkei Telecom

Speaking of Japan's market strength, the recent report of Dataquest Inc. estimated that the U.S. and Japanese semiconductor markets for 1986 would be $11 billion and $10.6 billion, respectively, and that Japan would surpass

the United States and become the best in the world. That which was never dreamed a decade ago is becoming a reality.

From 'Policy Friction' to 'Business Friction'

Japan's rise in position in the two aspects of production and market has, as a result, brought about the expansion of the "imbalance" in the trade incomings and outgoings, a "prime mover" of the U.S.-Japan semiconductor friction.

If we look at the trend of the IC trade between the United States and Japan, as shown in Figure 4, the United States recorded the excess exports over imports and Japan recorded the excess imports over exports throughout the 1970s. Although the United States had a positive balance of trade, the reason the first semiconductor friction took place in 1977 is that the Japanese group was rapidly increasing its share in terms of the 16K-bit DRAM whose production was on the rise at that time (the share in the peak year was about 40 percent). The U.S. side at this time makes the tariff barrier and the industrial policy of the Japanese group the object of attack and presses for correction. In this sense, the friction at this point had a strong aspect of policy friction.

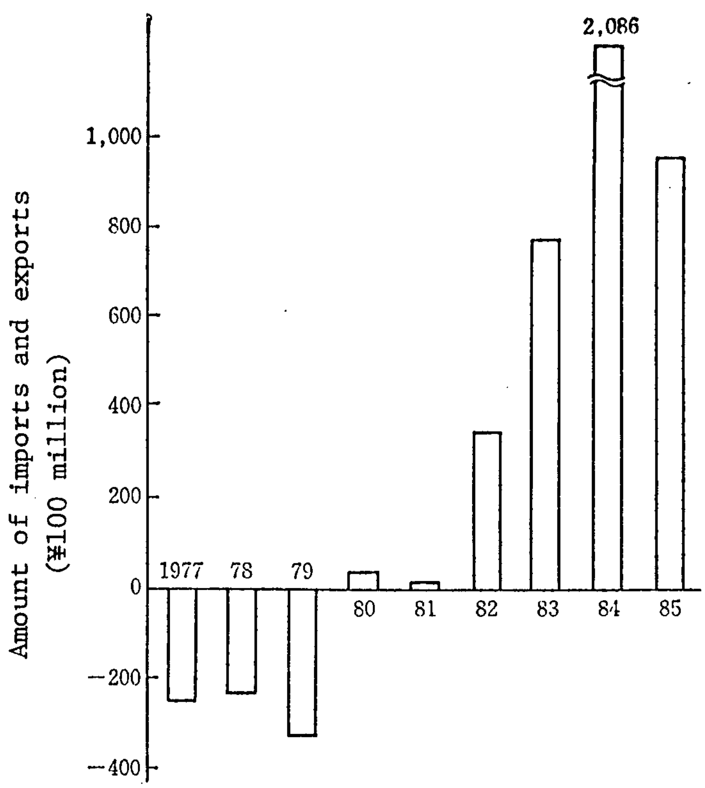


Figure 4. Trend of Japan's IC Trade Imports and Exports With the United States (Statistics of customs clearance by the Ministry of Finance)

With the arrival of the 1980s, the complete change leading to the basic trend of Japan's excess exports over imports and the United States' excess imports over exports has been made. The trade surplus of ¥2.8 billion in 1980 and of no more than ¥700 million in 1981 increased to ¥33.3 billion in 1982 and ¥76.7 billion in 1983 and attained as much as ¥208.6 billion in 1984. In 1985, the trade surplus was reduced by half, based on the stagnation in the U.S. market and the rekindling of friction; but even so, a trade surplus of ¥94.1 billion was recorded.

The friction between the United States and Japan in the 1980s, was caused for two reasons. Japan occupied 70 percent share in the 64K-bit DRAM market with the peaks recorded in 1982 and 1983. Another reason is connected with the development of the 256K bit market, the closed nature of the Japanese market and the dumping problem constitute two big themes. If we regard the friction of the first phase as a "policy" friction, the latest friction is "business" friction.

According to SIA data, the share of Japanese makers in the U.S. market increased from 2 percent in 1976 to 14.3 percent in 1984 (see Figure 5). On the contrary, the shares of the U.S. makers in the Japanese market remained at about 10 percent for the past 12 years (see Figure 6). The U.S. complains that in spite of the drastic changes in the environment such as the liberalization of trade and the reduction in the tariff rate, the U.S. share has not increased at all.

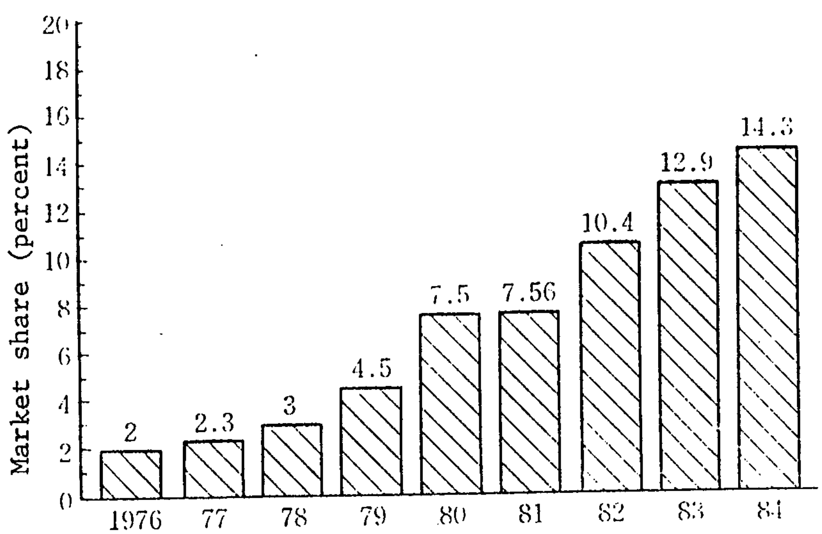


Figure 5. Shares of Japan's Semiconductor Makers in the U.S. Market (SIA [U.S. Semiconductor Industry Association])

Table 3 is based on MITI data but the share of U.S. makers in the Japanese market amounts to 19.1 percent if imports from the U.S.-affiliated makers in third countries and the in-country shipment by the U.S.-affiliated maker in Japan are included. On the other hand, the share of Japanese makers in the U.S. market is no more than 9.6 percent if production of in-house makers

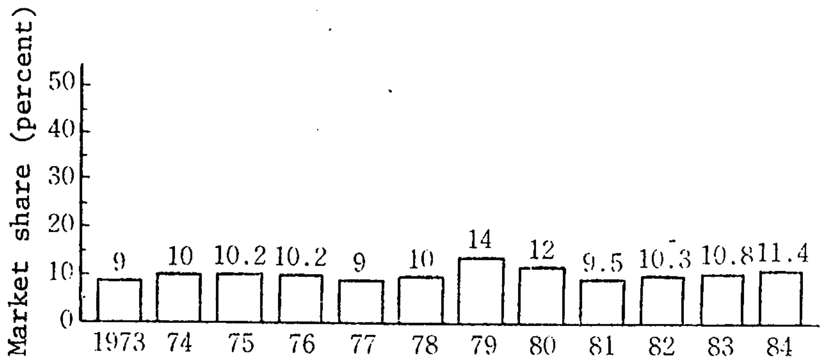


Figure 6. Shares of U.S. Semiconductor Makers in the Japanese Market (SIA)

Table 3. IC Supply-Demand Matrix Between U.S. and Japan

| | U.S. market | Japanese market | Total |
|---|---|---|---|
| Production by U.S.-affiliated enterprises | 14,366 ( 90.1) | 1,023 ( 19.1) | 15,389 |
| For internal consumption | 4,615 ( 28.9) | 281 ( 5.3) | 4,896 |
| For marketing to others | 9,751 ( 61.1) | 742 ( 13.9) | 10,493 |
| Production in the United States | 5,250 ( 32.9) | 331 ( 6.2) | 5,581 |
| Production in Japan | 22 ( 0.1) | 285 ( 5.3) | 307 |
| Production outside United States and Japan | 4,479 ( 28.1) | 126 ( 2.4) | 4,605 |
| Production by Japanese-affiliated enterprises | 1,531 ( 9.6) | 4,300 ( 80.4) | 5,831 |
| For internal consumption | 12 ( 0.1) | 577 ( 10.8) | 589 |
| For marketing to others | 1,519 ( 9.5) | 3,723 ( 69.6) | 5,242 |
| Production in the United States | 246 ( 1.5) | -- | 246 |
| Production in Japan | 1,267 ( 7.9) | 3,641 ( 68.1) | 4,908 |
| Production outside United States and Japan | 6 ( 0.0) | 82 ( 1.5) | 88 |
| Others | 50 ( 0.3) | 27 ( 0.5) | 77 |
| Total | 15,947 (100.0) | 5,350 (100.0) | 21,297 |

Note: Actual results in 1984; Unit is $1 million; figures in parentheses indicate the percentages of totals of United States and Japan; MITI survey.

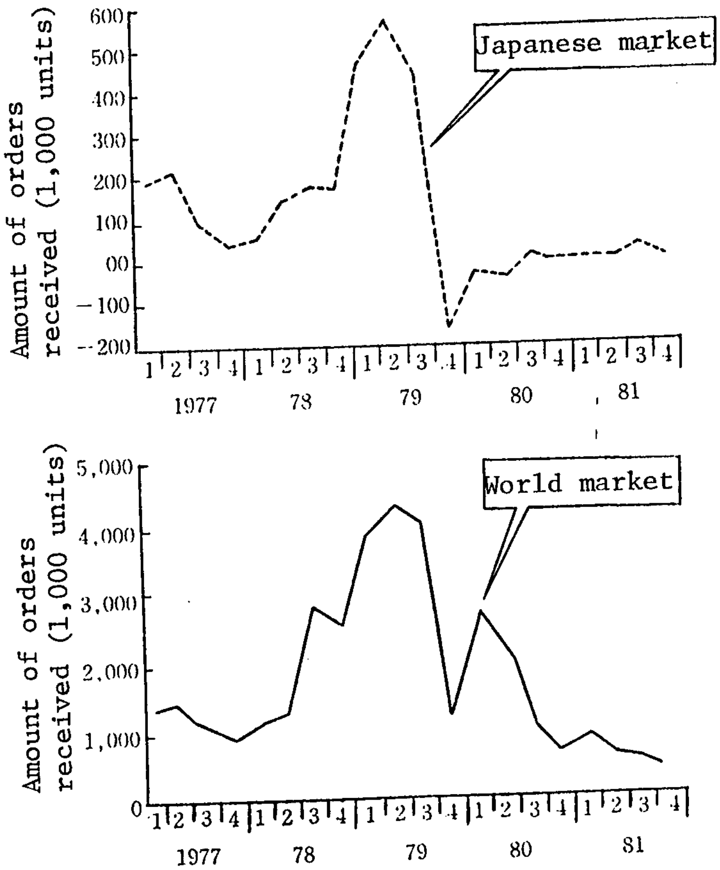


Figure 7. Trend of Estimated Amount of Orders Received for 8080 Microprocessors From U.S. Semiconductor Makers

such as IBM and AT&T are included. If we accept the logic of the U.S. side that the "market share" is a scale to indicate the rate of openness of the market, we can say that the U.S. market is rather more closed.

## U.S. and Japanese Business Urged To Reconsider Seriously

As I wrote in CHUO KORON in February [1986], we must say that the semiconductor friction between the United States and Japan is the inevitable consequence brought about by Japan holding the position of greatest semiconductor producer in the world. If we consider the fact that Japan's real ability will further increase from now on into the 1990s, it is certain that pressure from the U.S. side will further intensify. While coexisting with this friction, it will be important for us to work out countermeasures to ease the friction a little.

What is important is the fact that the semiconductor industry itself contains the extremely competitive primary factor. This is because the semiconductor industry is expected to experience an annual growth of about 20 percent toward

the mid-1990s. If so, competition is inevitable and without competition, progress will be retarded.

Friction is an inevitable accompaniment in an arena of competition. Just one word I would like to add is that as the growth of this industry is dramatic, this competition is not a "zero-sum game," and there is no inevitability to produce a "loser." There is an open path to "competitive coexistence."

In conclusion, I should like to say a few more words on the industries of both the United States and Japan.

Frankly speaking, misunderstanding and prejudice frequently creep into U.S. discussions on trade friction; distrust and threat become aggravated. Both the United States and Japan should see through the real substance of the situation and deal with it calmly.

With regard to the U.S. side, it should reflect on quality, reliability of delivery, and the service structure of its own products before it points out the closed nature of the Japanese market. I recall that a top manager of a certain major domestic maker said that "U.S. makers think only of their own logic and convenience, whether it be matters of quality control or delivery time. We are hesitant to buy from them even when we want to."

Also, the United States should recognize the great role played by the manufacturing branch and engage in its improvement. It may be impossible to shift to Japanese-style management, because of differences of culture, custom, and system between the United States and Japan. However, I wish the United States would learn Japan's strong points in production. U.S. semiconductor makers should also try to learn why U.S. major users are willing to use Japanese products.

Needless to say, there are points for the Japanese side to reflect on. The domestic maker is apt to make a huge investment based on capital strength partly because a big enterprise engages in the semiconductor business on a concurrent basis. Excess investment will lead to excess production, which may lead to excess exports. Although it is unfair to call Japan a "looter" of the memory market as a key figure with a major U.S. semiconductor firm has, the Japanese side has to understand the pain of the side that lost the market. An international division of labor, to include the newly industrializing countries (NICS), must be sought for the future.

Another point is that Japanese makers must give up imitation and the "free ride" on technology and make more risky efforts to conduct basic research and develop creativity. Through such fundamental steps, it may be possible for Japan to transform itself from a "great semiconductor 'production' country" to a "great semiconductor country" in a true sense of the word.

20,113/9365
CSO: 4306/5516

# EVALUATION METHODS OF ELECTRONIC MATERIALS DISCUSSED

Tokyo DENSHI KOGYO GEPPO in Japanese No 8, 1986 pp 44-48

[Article by Toshiaki Ikoma, professor, Research Institute for Industrial Technology, University of Tokyo]

[Text] 1. Introduction

The electronic materials evaluation method technical committee concluded a 2-year research study and reported its findings from the research study on 2-3 June by holding a briefing session, thereby completing its duty. It was a large committee having more than 40 members, and this is believed to have reflected the situation where great interest has come to be given to the unobtrusive field of evaluation method of electronic materials. Generally, the more advanced materials are, the more complicated evaluation technologies are and at the same time, the greater the importance of evaluation becomes. The abovementioned fact can be considered to be even an indication of the advancement in progress of electronic materials that holds the key to the development of present high technologies.

Electronic materials evaluation technologies are complicated and diversified. Electronic materials subject to evaluation also vary in type, such as semiconductors, metals, ceramics, polymers, and biomaterials. Further, even the evaluation technologies themselves vary, such as electrical technique, optical technique, chemical analysis, and physical analysis; the scale of equipment, too, varies from a simple one like voltmeter and ammeter to what uses SOR (synchrotron orbit radiation), which exceeds ¥10 billion in cost. Consequently, the subjects and methods of research also were extensive, and it was impossible to completely cover them in 2 years. Thus, this committee limited the research fields according to the specialized fields and the subjects of interest of its members.

In the initial year, the committee conducted research on the present situation and the problematical points of materials and evaluation technologies as widely as possible. In the second year, it conducted intensive research, including the future trend, by limiting the research fields. In the initial year, the committee conducted research on silicon, GaAs, organic materials, instrumental analysis, and electrical and optical evaluation methods. In the final year, it conducted research on the evaluation technology of Si LSI and compound semiconductor crystal devices. As the evaluation technology, it

conducted priority research particularly on the composite evaluation technology and the newest technique that becomes an important evaluation technology toward the future.

In this report, the committee outlines its findings mainly for this year.

## 2. Evaluation Technology in Si LSI Crystal Device Process

### (1) Silicon crystal

With the fining and the high integration of LSI, the quality improvement of silicon single crystal is being called for. At present, a silicon of no line-defect and large diameter ($\sim$ 8-inch diameter) has been realized. What is now called in question is the evaluation of electrically inactive impurities such as oxygen, carbon, and nitrogen, and point defect, and the evaluation of microscopic defect caused by process. Further, in addition to bulk crystal, epitaxial film has come to be used not only for bipolar LSI but also for MOS LSI in recent years. For epitaxial layer, evaluation of the lifetime and the planar and circular uniformity of impurity concentration profile is important, and mainly electrical technique is being adopted for it.

Oxygen in silicon changes its form by heat treatment, and sometimes makes a cluster and becomes electrically active. Further, since the bend of wafer due to heat distortion, etc., varies with the oxygen content, oxygen is an important impurity, and therefore, it is necessary to accurately measure its concentration. Usually, the infrared absorption method by localized oscillation mode is in use. For seeking its concentration, it is necessary to know the conversion coefficient between the amount of infrared absorption and the concentration, and thus its standardization is now being pushed. The infrared absorption method can measure only oxygen in a specific state (in the position of substitution, for instance). For measuring the whole amount, SIMS (secondary ion mass spectroscopy) and activation analysis are used. As for a very small amount of oxygen, the latter is reliable.

Not only to oxygen, the same evaluation method is applicable also to carbon and nitrogen.

For the measurement of microscopic defect, chemical etching, X-ray topography, and infrared scatter tomography are effective. For a further detailed observation, TEM [Transmission Electron Microscope] is used, but this is problematical as the evaluation method in that there is a need to become skillful in the preparation of sample sections.

The surface evenness becomes increasingly important with the fining of LSI. Optical technique and mechanical technique are often used usually, but a reflecting electron microscope (REM) using TME has been developed recently. REM is a method to observe the topograph by diffraction spotting of the RHEED pattern. By observing the REM image, minute unevenness of the silicon surface can be directly observed. As an example, on the silicon wafer surface, the existence of unevenness of a height of 12-16 Å and a cycle of 0.2-0.5 μm has been reported.

(2) Evaluation technology in IC process

With the fining of VLSI, there come the formation of shallow junction, the formation of low-resistance contact, and the thinning of gate oxide film as the main tendencies. Evaluation technologies also must be improved correspondingly with these tendencies.

First, for the measurement of the distribution of impurities of the shallow junction, SIMS is used. In this case, there is a fear that particularly by spattering, the initial distribution near the surface changes. Meanwhile, a combination of activation analysis by thermal neutron and anodic oxidation etching is also effective.

For the contact, high melting-point metals and their silicides are used, but a method combining electrical measurement, etc. of contact resistance and the like and metallurgical observation (scanning electron microscope (SEM) and TEM) is effective for knowing the microstructure of contact. Particularly, the observation of cross-sectional lattice image of the interface of silicon and silicide has recently been actively conducted.

Next, the problematical points and evaluation technologies attendant upon the thinning of gate oxide film and the fining of devices are mentioned.

When the gate oxide film becomes about 100 Å, the dielectric strength and the long-term reliability come into question. Particularly, the reduction of defects such as pinholes, and weak spots in the oxide film is necessary for improving the yield. The formation of these defects of the oxide film has connection with the property of substrate silicon.

In the thin oxide film with no defect, the state of dielectric breakdown varies with the lapse of time, and the TDDB (Time Dependent Dielectric Breakdown) method is used for evaluating the reliability.

With the fining of devices, large-energy electron and hole are injected into the oxide film, and this causes the instability of operation. This instability has a connection with the trap in the oxide film, and the evaluation of the trap is important.

(3) Inspection and evaluation of devices after IC process

The inspection after the completion of LSI is the final-stage evaluation technology in a seriesof IC processes, and is thus most important. As LSI becomes complicated, inspection technologies also become complicated and extensive. In recent years, a laser probe tester EB (electron beam) tester, which can conduct noncontacting and nondestructive test of complicated circuit, has been developed.

## 3. Evaluation Technology in Compound Semiconductor Device Process

### (1) Bulk crystal

Half-insulating GaAs crystal is now under active development and research as an LSI substrate, and its evaluation technologies required are also diverse. Among others, trace impurity analysis technology and nonstoichiometric evaluation technology are important. Particularly, the existence of excess As has been assumed from the precision measurement of the lattice constant, and its correlation with the distribution of EL 2 and the dispersion of FET threshold voltage has been clarified.

Since EL 2, the main electron trap in GaAs, is closely related to the manufacturing process of GaAs LSI, its control and the elucidation of what it really is are an urgent need. Mapping technology of EL 2 and line-defect distribution, striation, etc., also are being variously contrived, and correlations of the distribution of these defects and the FET characteristics also are becoming clear.

### (2) Epitaxial crystal

Since epitaxial crystal forms the active layer of laser and FET, etc., the upgrading of its quality is called for. Its purity depends on the raw gas or raw metal used, and thus trace impurity analysis technology is important. As such analysis technologies, atomic absorption analysis, ICP emission spectroscopy, spark ion source solid mass spectroscopy, etc., are in use.

The in-situ (in its original place) evaluation during the epitaxial growth also is important from the aspects of elucidation of the growth mechanism and precision control of the film thickness, but in other than the growth in high vacuum like MBE (molecular-beam epitaxy), equipment is not necessarily developing well partly because of its difficulty.

In MBE, the surface evenness and the film thickness can be controlled by using the oscillation phenomenon of RHEED. In the epitaxial growth using gas, monitoring by ellipsometry and Raman spectroscopy is being conducted.

A new method using TEM has been contrived for microscopic component analysis of the epitaxial layer. By using this, component analysis of ultrathin film in the superlattice has become possible. Besides, ordinary electrical and optical evaluation methods are being widely used for the evaluation of the epitaxial layer.

### (3) Device process

For manufacturing GaAs MESFET, ion implantation and annealing are used. For the evaluation of ion-implanted impurities and defects, SIMS (Secondary Ion Mass Spectroscopy), RBS (Rutherford Backscattering Spectroscopy), and AES (Auger Electron Spectroscopy) are used. Also, as the electrical and optical evaluation methods of defects, DLTS (Deep Level Transient Spectroscopy), and PL (Photo Luminescence) are in use. The evaluation process is conducted

eventually by whether the characteristics of manufactured devices have required performances or not. For the manufacture of LSI, however, the dispersion of threshold voltage in the wafer plane is the greatest problem, and thus its research using TEG is being actively conducted. Particularly, in the In-doped no line-defect crystal, the dispersion of threshold voltage is small, and thus it can be said that we have moved a step nearer to the practical use of GaAs LSI.

## 4. Trend and Future Image of Composite Evaluation Method

The composite evaluation method has two aspects: combined evaluation techniques and simultaneous evaluation during the process.

### (1) Composite analysis

For surface analysis, it has become possible to conduct in one chamber various analysis techniques in ultrahigh vacuum. For instance, a system simultaneously incorporating such techniques as AES, XPS, UPS, and ELS is being marketed. Further, a system connecting these techniques with MBE equipment in ultrahigh vacuum can also be used.

Composite evaluation technology using mainly the high-resolution TEM also is in widespread use. Even a total TEM system using TEM wherein CL (cathode luminescence), AES, ELS, X-ray analysis (EDX), etc., have been incorporated is being marketed.

Besides, for the composite evaluation of crystal defects, a method combining scanning TEM (STEM) and scanning DLTS (SDLTS), CL, and further IBIC (induced current) mode of SEM is under research. By this, the microscopic distribution of point defects can be measured.

### (2) In-situ evaluation of process

As has been mentioned above, since MBE allows simultaneous incorporation of surface analyzer, it is advantageous for the control of film thickness and the elucidation of growth mechanism. As the combination like this, MBE + RHEED, MBE + AES, and CVD + LIF (Laser Induced Fluorescence), CVD + infrared absorption spectrum, PCVD + emission spectral analysis, mass spectroscopy, and Raman spectroscopy, etc., can be cited. These in-situ evaluation technologies are still insufficient in their present situation; hereafter, research on various in-situ evaluation methods for the elucidation of growth mechanism and the design data for the reactor should probably be conducted.

The dry etching is being widely used in Si LSI, but the in-situ monitoring method is important. That monitoring method is divided into two types of plasma measurement to diagnose plasma itself and sample measurement to monitor the state of sample. These are very important, and therefore are listed together in Table 1.

Table 1. Monitoring Method in Dry Etching Process

| Classification | Measurement and detection method | | | Measurement and detection subject (physical quantity) | Equipment* |
|---|---|---|---|---|---|
| Plasma measurement method | Spectral analysis | Optical | Plasma emission method (ultraviolet, visible) | Chemical species in excitation state | △ |
| | | | Infrared absorption method | Chemical species in ground state | ○ |
| | | Laser | Laser absorption method | Chemical species in ground state | ○ |
| | | | LIF (laser induced fluorescence) | Chemical species in excitation state | ○ |
| | | | CARS (coherent antistokes spectrometry/scattering) | Chemical species in excitation state | ◎ |
| | | Electric wave | Microwave method | Electron density | ○ |
| | | | EPR (electron paramagnetic resonance) | Radical | ◎ |
| | Mass spectroscopy | | Ion mass spectroscopy | Charged particle in plasma | △ |
| | | | Electron impact (EI) ionization method | Neutral particle | ○ |
| | | | Photoionization method | Neutral particle | ○ |
| | Impedance (electrical) method | | Electrostatic probing method | Charged particle (electron) | △ |
| | | | Discharge (RF) voltage method | Charged particle | △ |
| | | | Photogalvanic spectroscopy | Neutral particle | ○ |
| Sample measurement method | Laser spectroscopic analysis | | Laser-beam interference method | Film thickness, etching depth | ○ |
| | | | Ellipsometry | Film thickness, etching depth | ○ |
| | | | Grating (diffracted light) method | Film thickness, etching depth | ○ |
| | Others | | Sample temperature measurement method | Temperature | △ |
| | | | Chemical luminescence method | Fluorescence | △ |

* △ Only detector system ( $\boxed{\text{working chamber}}$ → ◁ )

○ , ◎ (expensive): Excitation/irradiation system + detector system (▶ → $\boxed{\text{working chamber}}$ → ◁ )

## 5. Trend and Feasibility of the Newest and Future Technologies

With the advancement of electronic devices, for the evaluation methods, too, technologies for acquiring trace-quantity and minute information in more microscopic regions have become necessary. Here in this portion, some of the newest measurement technologies are picked up and described.

### (1) Utilization of synchrotron radiation (SOR)

SOR has such characteristics as 1) high luminance, 2) white light ($10^{-1}\sim10^{4}$Å) from X-ray to infrared region, 3) stability of beam, 4) parallel light, and 5) polarization trait, and provides very effective electromagnetic energy beams for the evaluation of materials. By using this, the hitherto impracticable measurement methods have become practicable.

Since topography can be taken in a short time by using the property of high luminance of radiation, the observation of such dynamic processes as formation of line defect, melting and congealing processes, and phase change has become possible. Further, by EXAFS of mixed-crystal semiconductor, the changes, etc., of the localized bond length have been clarified. Also, in the fluorescent X-ray analysis as the microanalysis method, the detection limit up to ppb has been obtained by using SOR.

### (2) Microanalysis method

With the purification of electronic materials, the detection of trace impurities has become necessary. The detection limit also has been improved, and in some impurities, up to the concentration of less than 10 ppb can be detected. The effective methods as microanalysis are 1) fluorophotometry ($\alpha$ ray emitter elements such as U and Th), 2) ion chromatography (Cl, Na, etc.), 3) SIMS, 4) mass spectroscopy, FT-IR (gas, organic matter), 5) charged particle activation analysis (light element), etc.

### (3) Microscopy

Electron microscopes have come to have an improved resolution, and TEM has now developed to the extent of enabling the observation of lattice image, but it is worth noting that a system having a resolution of 8 Å by using the scanning electron microscope has come to be marketed.

Further, for the observation of the unevenness of Å order of the surface, a scanning tunnel microscope (STM) has been invented. This is epochal. By using this, it has become possible to observe the form of one atom in reality.

Further, an X-ray microscope also is now under development, which has advantages compared with the optical microscope, the resolution is higher, and the transmission image of thicker samples is obtainable.

Only specialists as yet are able to use these new evaluation technologies, but it is hoped that progress will bring the day when they are used as general evaluation technologies.

## Conclusion

The foregoing is a summary of the findings of 2 years of research of the evaluation method committee. The evaluation method of electronic materials is a technology belonging to the so-called interdisciplinary region that involves various expertise. Thus it is presumed to be a place where only persons having deep basic knowledge and a wide range of interests can take an active part. This committee also is believed to have fully effectively functioned as a place where it is possible to become acquainted with people of wideranging specialized fields.

The author thanks committee members, including Shintaro Miyazawa and Tsugunori Okumura, for their cooperation.

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## UPDATE ON DEVELOPMENTS IN METALS, CERAMICS, PLASTICS

Tokyo NIKKO MATERIALS in Japanese in Sep 86 pp 20-26

[Text] Metals

### Active Metal Is Melted and Stirred by Electromagnetic Force of Vacuum Induction Smelting Furnace

Shinko Electric Co., Ltd., has developed a vacuum induction smelting furnace for high purity smelting of active metals such as titanium and aluminum. A melting and stirring system based on electromagnetic force is introduced in this furnace.

Generally speaking, the vacuum smelting furnace is excellent in degassing effect, and is used to smelt active metals such as titanium and aluminum which are easily oxidized by the reaction with oxygen or nitrogen. Also, it is widely used to smelt metals for aircraft which require the high reliability, and for high purity metals for semiconductors.

This new furnace consists of vacuum tank, vacuum exhaust system, smelting furnace, inverter power supply, and control devices. It possesses the following features: 1) the smelting speed is high, because metals are directly heated and smelted by induced current; and 2) it is excellent in reliability and operation because it has an overall monitoring system.

The company plans to serialize this new furnace as a system which can cope with metals of 10 to 1,000 kg calculated in terms of iron. Also, the company will install a model plant which can handle metals of 50 kg in terms of iron in the company's Ise Plant, and will open the model plant to users.

### The Furukawa Electric Co., Ltd., Cooperates With Raychem Corp. in the United States in Shape Memory Alloy

The Furukawa Electric Co., Ltd., has signed a contract for the company to cooperate with Raychem Corp. in the United States in research, application development, and the business of shape memory alloys and superelastic alloys.

The shape memory alloy possesses a characteristic by which even if it is deformed by force applied to it, it will memorize its original shape, and when it is at a certain temperature, will naturally return it to its original

shape. This characteris.ic was discovered at the Ordnance Research Institute of the U.S. Navy in 1963. Raychem Corp. started developing materials based on nickel-titanium alloys and alloys based on copper in the mid-1960s, and put them to practical use in a high pressure oil hydraulic piping coupling for jet airplanes in 1969. Since that time, the company has been manufacturing fasteners and connectors for microelectronics.

On the other hand, the Furukawa Electric Co., Ltd., started conducting research on a nickel-titanium alloy in 1963, and commercialized the alloy as a frame for glasses in 1981. Subsequently, the company has developed application products in the field of electric appliances, automobile, and medicine, and is proud of the overwhelming results of this development work in Japan.

At present, interest in the application of shape memory alloys is increasing worldwide. It is expected that this cooperation agreement will contribute to remarkable developments in the application of these alloys.

## High-Speed Rotation Is Realized by Adopting a New Bearing in Ultrahigh Performance Turbo Molecular Pumps

MHI (Mitsubishi Heavy Industries, Ltd.) has developed four kinds of ultahigh performance turbo molecular pumps with excellent light gas exhaust characteristics.

The bearings of these new pumps consist of pivot bearings and magnetic bearings employing a permanent magnet. High-speed rotation has been realized by using the new bearing in these pumps. That is, the speed of the PT-300 with an exhaust speed of 300 liters per second is about 60,000 revolutions per minute, and that of the PT-1500 with an exhaust speed of 1,500 liters per second is about 30,000 revolutions per minute.

This high-speed rotation has sharply enhanced the exhaust performance. The compression ration is more than $10^5$ for various gases (more than $10^8$ for nitrogen gas). It is extremely difficult to exhaust gases with small molecules, such as hydrogen, and helium. But the new pumps have shown excellent performance in exhausting such gases. Also, it is said that these new pumps have an almost constant exhaust speed for various gases in wide vacuum areas.

Aluminum alloy is used in the body of the new pumps to reduce weight. The adoption of the new bearing in these pumps has achieved the following: 1) Clean ultrahigh vacuum conditions are created with almost no evaporation of oil; 2) the degree of vibration and noise is extremely low; and 3) it has a long life and is maintenance-free.

In addition to the two kinds of the new turbo molecular pumps, also available are the PT-500 with an exhaust speed of 500 liters per second and PT-3000 with an exhaust speed of 3,000 liters per second.

## Ceramics

### Establishment of Mass Production Technology for FRC Composed of Aramid and Vinylon

The Shimizu Construction Co., Ltd., has established a technology to mass produce hybrid type FRC (fiber reinforced concrete) composed of vinylon fiber and aramid fiber which have already been developed. The company made first practical use of the hybrid type FRC in the external wall materials for a new research building being presently constructed at the company's Research Laboratory at Etchujima in Tokyo.

This hybrid type FRC is manufactured by laying vinylon fiber mesh on concrete, placing cement containing aramid short fibers on the concrete, and laying vinylon fiber mesh thereon. It is a high-strength concrete with a bending strength which is four to five times that of normal concrete.

The amount of fiber contained in the usual FRC is high, being 3 to 4 percent (by volume), while that of the new FRC is low, being less than 1 percent, because the necessary strength can be obtained by skillfully combining expensive aramid fibers with inexpensive vinylon fiber meshes. For this reason, it is said that the cost can be held below the conventional cost.

### Refractory Bricks Made of Fine Ceramics Are Hot-Repaired by Using Plasma Spraying Method

Sumitomo Metal Industries, Ltd., has developed a new method of cementing refractory bricks made of fine ceramics. This is a hot-repair method based on the plasma spraying method, and has the following features: 1) the cementing work can be carried out even when the interstice is 5 mm or more; and 2) the strength of the cemented sections is about 1.5 times that of the base material.

The walls of furnaces such as high temperature steel furnaces, are fairly often damaged during operations. Up to now, such damaged walls have been repaired by using a flame spraying method based on the addition of water. But this method has a problem in that even if damaged refractory bricks are repaired, they will immediately be broken, because the cementable interstice is narrow, being 2 mm on average.

The company has used the following method. That is, an interstice with a width of 5 mm, just like a joint, is made on alumina-based refractory bricks containing about 5 percent silicon dioxide, these bricks are arranged in order, and argon gas is poured into each interstice of the bricks by using a plasma arc spray gun. This method is called, "Plasma Spray Treatment."

The sprayed material is an alumina-based refractory raw material powder, and consists of particles of a fixed size; that is, it has the same composition as the refractory bricks. The melting point of silicon dioxide is 1,713°C and that of alumina is 2,050°C, while that of argon plasma is high, being 3,000°C to 5,000°C. By using this high melting point, the material to be

sprayed is melted instantaneously, and is sprayed on the joints, and coats them with this material. The coating portions are amorphous films which will grow to fill up the joints.

It has been confirmed that when joints are heated to a temperature of 800°C, their bending strength will be about 120 $kg/cm^2$, which is about 1.5 times that of normal refractory bricks.

Surgical Carbon Dioxide Laser System Based on Optical Fiber Method

Matsushita Industrial Equipment Co., Ltd., has developed a surgical carbon dioxide laser system based on an optical fiber method in collaboration with Matsushita Electric Industrial Co., Ltd., and Matsushita Research Institute Tokyo Inc., and has started selling it. It is compact and is excellent in operation.

Generally, a mirror-articulated system is used in an optical guideway which introduces laser beams emitted from a surgical carbon dioxide laser system, but it is difficult to operate. Also, generally, the carbon dioxide laser oscillating tube is based on a system in which a gas composed of carbon dioxide, nitrogen, and helium is supplied at a constant pressure in a laser tube and is exhausted to the outside. However, it requires a gas cylinder and an exhaust unit. In addition, the gas cylinder must be replaced with a new one.

When both end faces of optical fibers are coated with antireflection membranes, the new laser scalpel will bring about a transmission efficiency of 82 percent. Also, Matsushita Industrial Equipment Co., Ltd., has developed a sealing type carbon dioxide laser oscillating tube which can continuously generate laser oscillations with an output of 45 watts for 1,000 hours or more. As a result, the device has been simplified and its maintenance enhanced. In addition, the company has achieved a guide beam system in which the irradiated point can be readily adjusted to a focus of carbon dioxide laser beams by mating the focus with a position at which the two guided beams (helium-neon laser beams) intersect.

High Purity Zirconia Fine Particles Manufactured at Low Cost by Desiliconizing Carbon Under Reduced Pressure

Kawasaki Steel Corp. has developed a method to manufacture high purity zirconia fine particles, and will branch out into a business of fine ceramics on a full scale. These high purity zirconia fine particles are manufactured from zircon by desiliconizing carbon under reduced pressure.

The new method means that after moldings are manufactured from the mixed powder and are heat-treated in a vacuum (Tanmann) furnace at a temperature of 1,105°K to 2,000°K and a reduced pressure of 100 pA, they are oxidized. This zircon powder contains more than 99 percent zirconia and silica. Such zircon powder, carbon, and the necessary stabilizer are added to the mixed powder.

Such moldings can be desiliconized efficiently by heat treating them at a reduced pressure, and they can be changed to zirconia single phase by oxidization. In addition, it is said to be possible to manufacture the partially stabilized zirconia fine particles by heat treating, desiliconizing and simultaneously solid dissolving the mixed moldings to which stabilizers such as calcia, and yttria have been added.

The electromelting and desiliconizing method and the alkali melting method are conventional methods to manufacture zirconia powder. The former method is low cost, but does not produce high purity zirconia powder. In contrast, the latter method produces high-strength and ultrafine zirconia powder, but is high cost.

According to the results of tests conducted by the company, after the 99.7 percent-plus powder, equivalent to that based on the alkali melting method, is oxidized, its particle size will be reduced to less than 1 micron by pulverizing the powder which previously had a particle size of less than 5 microns. Also, the company has developed a zirconia with an average particle size of 0.5 micron. This zirconia is partially stabilized by adding yttria for high-strength fine ceramics.

Commercialization of Ternary Ceramics Porous Body in High-Temperature Filter Media

Kobe Steel, Ltd., has developed a new filter by processing ternary ceramics in porous bodies and has started selling it as a high-temperature filter medium by the brand name "Acto-Thermic."

That is, the new filter is manufactured by forming cordierite (composed of magnesium peroxide, 2-alumina, and 5-silicon dioxide) in the shape of a thin line and by processing the formed cordierite in the shape of instant Chinese noodles. There is no porosity in the thin line, but when the thin line is formed in the shape of dried disk noodles, porosities will occur in the space between laminated lines.

Cordierite is a ceramic excellent in high-temperature resistance (melting point: 1,350°C, and has a small coefficient of thermal expansion ($1.5 \times 10^{-6}$) which is one-tenth that of steel. Cordierite is suitable for separating impurities such as copper and aluminum with melting temperatures of 750°C to 1,000°C. Particularly, it shows the very high wettability against such oxides as calcium, iron, magnesium, and silicon which are contained in the impurities being melted.

It is devised so that the impurities in solution will adhere as oxide films to the surface of the cordierite thin lines.

For example, 9.53 to 12.81 percent impurities are contained in the melted aluminum in a furnace, but when the new filter is applied to the melted aluminum, the impurities are reduced to 0.08 to 0.72 percent, showing a removal rate of more than 90 percent. Also, the maximum duration of use of the new filter is 30 days under conditions in which melted metal at the rate of 100 kgs/hr is poured into a furnace for 21 hours a day.

The price of the new filter with a diameter of 80 mm and a shape of dried noodles (50 cc) is ¥400-500.

## Hot Water Isotropic Pressure Laminator Uniformly Forms Ceramics

Nikkiso Co., Ltd., has developed and begun sales of a hot water isotropic pressure laminator used to contact-bond or to form semiconductor parts.

This new laminator is devised so that a pressure vessel is completely immersed in a hot water tank and ceramics are formed by circulating the hot water while keeping a constant temperature. It is also devised so that pressure is equally applied from all directions to the inside of the pressure vessel. Conventionally, only two or three sheets can be contact-bonded, but now about 20 sheets such as ceramics can be contact-bonded at one time by using the new laminator. Also, the accuracy of the products has been enhanced, and there has been no defective product (up to now, about 20 percent of all products were defective). In addition, the new laminator possesses the following features: 1) by heating the materials with hot water, processing work can be carried out at a lower pressure than that at normal temperatures; and 2) full automatic operation can be carried out by using computers.

The maximum pressure is 350 kg/cm$^2$ and the working temperature is up to 80°C. Materials with a maximum square of 17 cm can be treated.

## Fine Ceramics Are Uniformly Electroplated on Aluminum

Nippon Light Metal Co., Ltd., has commercialized a technology by which fine ceramics are uniformly electroplated on the surface of aluminum and has succeeded in sharply enhancing the wear resistance of aluminum. This is called "Dispersion Nickel-Phosphorous Plating Technology" and was introduced from the Suzuki Motor Co., Ltd. Products manufactured using the technology are used in engine cylinders of two-wheeled vehicles because these products are superior in wear resistance to those made of hard chromium.

Speaking in detail, this dispersion nickel-phosphorous plating technology means that a dispersion agent containing fine ceramics is put in a nickel sulfamate bath to which phosphorus is added, and ultrafine ceramics are eutectoid-dispersed in nickel layers electrodeposited by using an electroplating method. The use of the new technology has increased the wear, heat, chemical, and sliding resistances of the surface of metals. When silicon carbide is used as a dispersion agent, the wear resistance increases, and when hexagonal boron nitride is used as a dispersion agent, the lubricity and antiseizing effect increases.

When metals are plated by the new technology, internal stress will be small in the plated layers, and peeling will hardly occur on the plated metals. However, the company has increased the adhesion by activating the surfaces of metals and making them uneven by pretreatment.

## Easy Bonding of Ceramics and High-Strength Dissimilar Material

Nikkoshi Co., Ltd., has developed, by a simple process, a technology for bonding ceramics and dissimilar material with a strength about 15 times that of materials manufactured using conventional methods.

This new technology, called "Dispersion Bonding," is used to bond dissimilar materials such as ceramics and metal, and ceramics and plastics, as well as ceramics and ceramics. This bonding process consists of inserting material based on aluminum, silicon, or nickel into the bonding part and these bonding sections are heated and pressurized in a vacuum furnace in accordance with the conditions of the temperature used.

Because of future diversification, it has been expected that ceramics will be used as devices and systems by combining these ceramics with each other or by combining them with dissimilar materials. However, their development has been limited because no high-reliability bonding technology had been established.

When the new technology is used, materials with a greatly different coefficient of thermal expansion can be bonded readily with each other, and it is possible to cement oxide ceramics with alumina, and nonoxide ceramics such as silicon carbide and silicon nitride. The bonding strength is high, being 30 $kg/mm^2$, which is about 15 times greater than that achieved by conventional methods. The temperature used will not be reduced to 300°C. Also, when the quality of inserted materials is changed, the temperature used will be raised up to 700°C.

# Plastics

## Test Facility for Mass Producing Rare-Earth Plastic Magnets

Sumitomo Metal Mining Co., Ltd., has constructed a test facility for mass producing rare-earth plastics in a plant site of its wholly owned subsidiary, Taihei Metal Industries, Co., Ltd., and has started operating it on an experimental basis. The plant site is located in Sagamihara City, Kanagawa Prefecture. Also, Sumitomo Metal Mining Co., Ltd., will complete an intermediate scale test facility in the above plant site by November, and will establish a multikind small-lot production system. This facility will produce the precision casting parts and injection molding powder metallurgy for precision parts.

The rare-earth plastic magnet which has been developed by Sumitomo Metal Mining Co., Ltd., is called "Wellmax" (brand name), and is based on cobalt-samarium. That is, after the samarium oxide and cobalt powder are reduced and burned, nylon, which is a binder, and plastics are mixed with each other, and the injection molding process is carried out while applying the required number of magnetic poles and an equal number of magnetic fields. Compared to sintering type rare-earth magnets, rare-earth plastic magnets have the stronger magnetic force and the advantage of light weight.

The injection molding powder metallurgy is called "Metamold" (brand name), and has both rare-earth plastic magnet and basic technology. This Metamold is manufactured in accordance with the following process. After metal powder is mixed with an organic substance binder, this mixture is injection-molded, and the binder is removed from the mixture to enhance its moldability. Subsequently, the mixture with no binder is burned. Up to now, when a binder is removed from such mixture, the mixture shrinks by 30 to 40 percent. For this reason, it had been regarded as difficult to use such a mixture with no binder in the field of precision parts. However, this difficulty has been solved in the Metamold and it is easy to process such materials as stainless steel in three-dimensions.

Toray Industries, Inc. and Phillips Co. (United States) in Joint Enterprise for Domestic Production of PPS Resin

On 1 July Toray Industries, Inc. established a joint concern called "Phillips Petroleum Toray Inc." capitalized equally by Toray Industries, Inc. and Phillips Co. in the United States. The purpose of this enterprise is to promote positively in Japan the domestic manufacture and sales of PPS (polyphenylene sulfide) resin which was developed by Phillips Petroleum Co. in the United States.

The capital of the new company is ¥6 billion and the head office is located in Kojimachi, Chiyoda-ku, Tokyo. Mr Junichi Kabe, former general manager of Toray Plastic Planning Department has taken office as president. A new plant with an annual base resin production capacity of 7,500 tons will be constructed at a cost of ¥10 billion in the Toray Tokai Plant located in Tokai City, Aichi Prefecture. The new plant will start manufacturing PPS resin on a full scale in December 1987.

The PPS resin is an engineering plastic excellent in heat resistance, chemical resistance, and flame retardance, and is used as a material to replace high-function insulators and various metals in many fields. Phillips Petroleum Co. in the United States developed the PPS resin in the 1960s, and at present, the company annually produces 6,000 tons of PPS resin at its plant in Texas. In Japan, Phillips Petroleum International, Ltd., has imported the PPS resin from the Phillips Petroleum Co. In addition, Toray Industries, Inc. and Phillips Co. in the United States have carried out the development of a technology for improving the PPS resin since they established a joint development company in 1983.

Last year, the scale of the market for PPS resin in Japan was less than 2,000 tons. It is anticipated that the demand for PPS resin in Japan will increase to 5,000 tons in the future. Toray Industries, Inc. has decided to produce PPS resin domestically in Japan in collaboration with Phillips Co. because it is anticipated that world demand will increase up to 15,000 tons in 5 years.

Compact Injection Molding Machine Which Can Be Produced on Assembly Lines

Kawaguchi, Ltd., has developed a compact injection molding machine, and has started selling it by the brand name of "K JECTION-5." This new machine can

be readily incorporated in assembly lines, and can mold materials and can supply them as necessary parts on the spot.

In recent years, the industrial world related to small resin molding parts has inevitably taken up a multikind small-lot production system, and it has been of urgent necessity for the industrial world to automate production. However, small molding machines on the market now possess only the function to mold. For this reason, when insert parts are inserted into these molding machines or moldings are directly supplied to assembly lines, there are production and space problems.

The K JECTION-5 possesses the basic capability as a molding machine, and it is designed so that it can cope with wide system configurations as an FA (factory automation) component. The features of the new machine are as follows: 1) it requires a small space for its installation, and can be freely incorporated in assembly lines; 2) it does not require any oil control or water system because it is driven by electricity or air, and is readily transferred and installed in other places; 3) the molding work can be quickly started because the heating cylinder can be heated up completely in 5 or 6 minutes; and 4) it is possible to make system configurations with a high degree of freedom because the control box is separated from the body. The price is ¥3.2 million.

System for Predicting Durability of Vibration Free Rubber and Vibration Free Rubber With a Life of 60 Years

Bridgestone Corp. has established a system called "LALDA" (Life Assurance with Large Deformation Analysis), and has developed a vibration free rubber, with a durability of 60 years, called "Multirubber Bearing" in accordance with a new durability design method based on this system. The system is used to predict the overall durability of vibration free rubber which is a component of the vibration free structure.

The vibration free structure attenuates the earthquake impact propagated to large equipment and buildings by one-third to one-fifth, and protects these buildings and their furnishings from earthquake damage. The vibration free rubber absorbs the earthquake impact in this structure.

The LALDA is used to simulate how vibration free rubber is deformed by long-term use and by earthquakes and to simulate to what position and to what extent local stress is generated by this deformation. By using LALDA to predict the successive changes in the material characteristics of rubber exposed to the atmosphere, the company has succeeded in comprehensively predicting the durability of vibration free rubber. As a result, it has become possible to predict the performance and durability of vibration free rubber used over a long period of time and to design the entirety of architectural structures with consideration to the safety, performance, and durability based on this prediction.

The Multirubber Bearing is designed on the basis of LALDA, and is constructed with laminar thin rubber and copper plates. While applying a large load in

the perpendicular direction, it moves softly, by action of the rubber, in the horizontal direction. Also, ozone, deterioration, and radiation resistance have been enhanced by coating the external surface of the Multirubber Bearing with a newly developed specialty compound rubber to inhibit surface deterioration from the atmosphere.

Intermediate and Large Injection Molding Machines Enhance Energy Conservation and Productivity

MHI (Mitsubishi Heavy Industries, Ltd.) has developed and begun sales of five kinds of intermediate and large injection molding machines called "MG Series" which can cope with a multikind and small-lot production.

The MG Series are further developed model changes of the MF Series class with a mold clamping force of 350 to 850 tons. The productivity and operation of the new series have sharply been enhanced because these series have microcomputer controls as standard equipment. In addition, the price has been left the same as for the conventional MF Series.

All the MG Series are equipped with an automatic storage unit to memorize molding conditions, and this unit is controlled with a microcomputer. The operation preparing time can be reduced sharply because the MG Series can instantaneously reset optimum molding conditions even during operation. Also, an automatic color replacing circuit developed independently by the company is incorporated in the MG Series so that the replacement of colors and resins can be carried out in a short time. The high-quality stable molding work has been realized by adopting the straight hydraulic mold clamping system, high-rigidity panel, long L/D screw, and high-response logic valve from the MG Series.

In addition, energy and resource conservation has been realized by using a pressure match circuit with no energy loss and a mold clamping block circuit in the MG Series and by completely controlling the contamination of hydraulic oil.

The price of 350 MG with a mold clamping force of 350 tons is ¥33 million, and that of 850 MG with a mold clamping force of 850 tons is ¥83 million.

Multifunction and Long-Life Gland Packing Material

W.L. Gore & Associates Inc. in the United States and Japan Gore-Tex Inc. have developed a gland packing material called, "Gore-Tex GFO Fiber," and have started full-scale sales. This new material is excellent in chemical resistance, heat resistance, and thermal conductivity, and can sharply reduce the total cost.

The new packing material is manufactured by using an independent method. That is, graphite is fiberized and integrated completely in the Gore-Tex structure. Therefore, the Gore-Tex GFO Fiber has a fibrous structure in which PTFE (polytetrafluoroethylene) and graphite are completely integrated. The seal surface can be kept without any seizure in perfect condition at all times, because the

Gore-Tex GFO Fiber has excellent thermal conductivity and low-thermal expansibility which readily disperse heat. In addition, the new packing material possesses the following features: 1) it can be used at a high-peripheral speed of 20 m per second because it has the high-self-lubricity and is integrated with the lubricant; 2) it will neither wear any other substance nor damage any shaft and sleeve because it is flexible; and 3) it can be readily installed anywhere or be readily replaced because it is excellent in dimensional stability and will not be hardened by use.

It is said that a duration of use several times to tens of times that of conventional packing materials can be obtained thanks to the above features. The chemical resistance is pH 0 to 14 (except for strong oxidizing acid). The temperature of the heat resistance is minus 240°C to plus 260°C.

Also, the Gore-Tex GFO Fiber will be commercialized by Nippon Valqua Industries, Ltd., and Nichias Corp., and these companies expect to sell 30 tons of the Gore-Tex GFO Fiber in the first fiscal year.

Pretreatment Agent and Quick Setting Adhesive for Polyolefin

Toagosei Chemical Industry Co., Ltd., has put on the market an adhesive called "Aron Alpha PP Set." Up to now, it has been considered difficult to bond polyolefin such as polypropylene and polyethylene. But such polyolefin can be bonded intantaneously by using the Aron Alpha PP Set.

This set consists of a quick setting adhesive "Aron Alpha" and a fast drying special purpose pretreatment "Aron Polyprimer-D." It possesses the following features: 1) polypropylene and polyethylene, can be bonded instantaneously, as can EPT (ethylenepropylene-terpolymer) rubber, polyurethane, and nonrigid polyvinyl chloride; 2) according to the JIS (Japan Industrial Standards), the use of polypropylene will cause the break of materials with a thickness of 2 mm because of its strong adhesive power; 3) a small amount shows its power; and 4) it will not color materials to be bonded.

The price of the Aron Alpha PP Set including a spare is ¥2,800. The company is scheduled to sell it mainly in the fields of automobiles and weak currents and anticipates that the number of sets sold per month will be 10,000 for the time being.

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# RECENT DEVELOPMENTS IN PLASTICS, METALS, CERAMICS REPORTED

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[Text] Plastics

## Method of Manufacturing Jig Used To Test Tensile Fatigue Strength of Ferro-Concrete Reinforcement Bars

Sanyo Chemical Industries, Ltd., has developed a reinforcing method based on the use of adhesives and aluminum pipe under the leadership of Tadashige Kawai, lecturer at Nihon University, College of Industrial Technology. This is a method of manufacturing jigs used to conduct tests for tensile fatigue strength of reinforcements. Compared with conventional methods, this new method will bring about easy manufacture of such jigs at a lower cost.

In recent years, the need for reliability of reinforcements has increased in proportion to the increase in demand for the reinforcements, and there has been a tendency to demand basic data on fatigue resistance. When tests for tensile fatigue strength of reinforcements are conducted, it is necessary to reinforce the clamp of the jibs because the vibration force tends to concentrate in the clamp. In addition, when test reinforcements are installed without any change on a testing machine, the strength of the clamps will be lowered by the tightening force, and these test reinforcements may be cut readily. There are several methods to reinforce the clamps, but these methods have some problems such as high-cost, and they demand high skill.

The new method is as follows: 1) an aluminum pipe with a length of 20 to 30 centimeters is fitted onto the end of a reinforcement with a length of about 1 meter; 2) a structural adhesive based on special epoxy is put in the space between the aluminum pipe and reinforcement; and 3) the section covered with the aluminum pipe is pressurized with a press elliptically to form this section and to harden it statically. This structural adhesive is called "Unibon EP-1403," and has been newly developed by the company. This new method has the following features: 1) it requires neither special facilities nor high skill; 2) the cost is low, being 30 to 40 percent of the conventional cost; 3) the reinforcing effect is equal to or greater than conventional methods; and 4) no strain is generated in test reinforcements.

All-Plastic Acceleration Sensor Made

Mitsubishi Petrochemical Co., Ltd., has developed an all plastic acceleration sensor in which piezoelectric plastics are used, and has started selling it by the brand name of "Super G."

The acceleration sensor measures such movements as the vibration and sway of objects. It is expected that the acceleration sensor will be widely used to measure the vibration and sway of automobiles, vehicles, and various machines. Existing acceleration sensors made of piezoelectric ceramics are expensive, and have some problems in respect to freedom of usage.

In contrast to existing acceleration sensors, the Super G has the following features: 1) because it is made entirely of plastic, it has excellent shock resistance, is waterproof, and it can be easily handled; 2) compared with conventional acceleration sensors, a sharp reduction in cost of the Super G has been achieved, and it can be readily used in new fields; 3) it is optimum for measuring the vibration and sway of various engines and structures, because it can measure them up to a low frequency region; 4) it can measure the vibration of a single axis with high sensitivity and high accuracy because the horizontal and vertical separations are satisfactory; and 5) it can be readily installed because it is thin, compact, and lightweight.

The company has established a production system in its Yokkaichi Works. Initially the Yokkaichi Works will sell 5,000 to 10,000 Super Gs every month.

The price is ¥4,800. The company will distribute the Super G as a general-purpose acceleration sensor.

Portable Surface High-Resistance Meter

Mitsubishi Petrochemical Co., Ltd., has developed a surface ohmmeter, and has started selling it by the brand name "Hiresta." The Hiresta measures with high accuracy the surface resistance of materials used to prevent electrical charges and electrostatics.

Antistatic and destaticized films and plastics are used increasingly as parts and materials of OA (office automation) equipment such as electrostatic copying machines and facsimile machines. They are also used as parts and materials of bags, storage cases, and protective boxes which are used to prevent accidents caused by adhesion of dust or from sparks occurring in electronic parts such as ICs or LSIs.

The Hiresta is used to evaluate and inspect these parts and materials. It possesses the following features: 1) its measuring range is wide, being $1 \times 10^6$ to $9.999 \times 10^{12}$ ohms; 2) it is compact (217 x 229 x 84 mm), light weight (1,280 g), and portable; 3) large EF-type liquid crystals provide easy readout; and 4) it can be used to make measurements with single finger motion, because of its special-purpose probe.

The price is ¥285,000.

## Ferroelectric Liquid Crystal Compound Is Characterized by Clear Contrast

Dainippon Ink & Chemicals, Inc., has developed a ferroelectric liquid crystal compound called, "Chiral Smectic C Phase" in collaboration with Kawamura Physics and Chemistry Research Institute, and will start shipping it to electric and electronic manufacturers.

The smectic C phase in a typical ferroelectric liquid crystal has the major axis of liquid crystal molecules slope at a constant angle and the liquid crystal is wholly layered. When voltage is applied to the liquid crystal, the time necessary to change the direction of the liquid crystal molecules is less than one-millionth of a second. The smectic C phase has come into the limelight as a next-generation liquid crystal because this time is very fast compared to that of nematic phase crystals.

The kinds of liquid crystal compounds developed by the company exceed 200. All the liquid crystal compounds are based on aromatic esters included in the ester family. These compounds possess the following features: 1) the chiral smectic C phase shows the ferroelectricity (even when there is no external electric field, there is external polarization), and has a correspondingly wide temperature range; 2) the spontaneous polarization is large; 3) the viscosity is low; 4) the adhesiveness and pitch can be adjusted freely; and 5) the slope angle necessary for clear contrast is large. Therefore, it is said that the above compounds display their characteristics as indicator materials--excellent contrast, memory characteristics, bistability, and high-speed response properties (when the temperature is 25°C, the voltage is 20 volts, and the width between electrodes is 2 microns, the response speed is 200 microseconds). Also, the compounds can be colored freely because they are colorless.

For the time being, the company will further develop the new compounds for use in the high-speed printers and in large displays for computer terminals of 640 x 400 dots.

## Humidifying Power of Moisture Permeation Type Humidification Film Module Is Enhanced by Using Porous Plastic Film

Mitsubishi Electric Corp. has developed a humidification film module whose power is sharply enhanced by applying a moisture permeating film to a natural evaporating type humidification method.

In recent years, while compact dispersion systems have been the main air-conditioning systems for buildings, it is anticipated that these air-conditioning systems will be equipped with humidifiers in the near future. For this reason, the development has been expected of a humidifier module capable of ready incorporation in compact air-conditioners, and which can provide clean humidification without the occurrence of white powder, and such.

The company has developed a water-repellant and porous plastic film in collaboration with Mitsubishi Chemical Industries, Ltd. This film possesses a characteristic that it is permeable to water vapor, but is impermeable to

water. The company has completed a moisture permeation type humidification module whose moistening power is sharply enhanced by applying this moisture permeable film to natural evaporation type humidification equipment. The main features of this module are as follows: 1) it can humidify cleanly because calcium which has dissolved in water is not supplied to it; 2) the humidifying power is high because the moistening surface area is about eight and one-half times that of natural evaporation type humidification modules; and 3) by merely setting it in an air duct of an air conditioner, it will perform its humidifying function.

The company is planning to put it on the market as a product which can be mounted in a fan coil unit, a building air conditioner, or an external air treating unit.

Plastic and Metallic Bolt With High Strength and Excellent Corrosion Resistance

Bridgestone Corp. has developed a nut and bolt, and has started selling it by the brand name "Super Bolt." This Super Bolt has the strength equivalent to metal, and the corrosion and chemical resistance equivalent to plastics. In addition, it does not loosen because of vibration.

Mainly, iron or stainless steel nuts and bolts have been used up to now, but they have a problem of corrosion. For this reason, plastic bolts are used in parts which must be corrosion resistant, but these plastic bolts have the disadvantages of low strength, being one-fourth to one-sixth that of metallic bolts, and of high price.

The company has suceeded in developing the Super Bolt with both strength and corrosion resistance by combining the metal with engineering plastics. The price is almost the same as that for stainless steel bolts. In addition, the new bolt possesses the following features: 1) it has an excellent locking performance against vibration; 2) it is excellent in heat insulating properties, and has a dew condensation preventive effect; and 3) it can be used at a temperature range from 50 to 250°C depending on the kind of plastic.

It is available in seven sizes from M6 (thread diameter of 6 mm) to M24 (thread diameter of 24 mm).

Experimental Manufacture of Water Jet Cutter Used To Remove Burrs From Large Castings

Dengyosha Machine Works, Ltd., has completed test manufacture of a water jet clutter and will start taking orders for it this autumn. This water jet cutter can cut steel plates with a thickness of 70 mm at a speed of 100 mm per minute.

The water jet cutter consists of high-pressure pump, booster, cutting unit, control panel, abrasive feeding unit, and water supply unit. It is used to cut steel plate, sheet glass, and reinforced concrete by applying abrasive and water jetted from its nozzle into these materials. Synthetic diamonds are

used in the nozzle which has the most important function. The water jet cutter is devised so that water containing abrasive is jetted from the nozzle which has a diameter of 2 to 3 mm.

The maximum pressure is 3,000 $kgf/cm^2$, the flow rate is 9.4 liters per minute, and the output is 90 kw. In the future, the company plans to develop a robot to control the movement of the nozzle, and to complete two models (portable type and installed type) of water jet cutters.

The company expects that the demand for water jet cutters will be increased in the field of burr removal from large castings, the work of cutting and dismantling buildings, machine work, and such large projects as tunneling.

## Metals

### High-Resistance Alloy Which Can Be Used in High-Load Regions

MMC (Mitsubishi Metal Corp.) has developed a new tool material, and has started selling it by the brand name "High Resistance Alloy." This new material has the characteristics both of conventional cemented carbide and high-speed steel.

This high-resistance alloy has been developed so that it can cope with the expansion of high-load regions caused by an increase in the speed of the hot roller and the steel bar roller. It is manufactured by using a powder metallurgy method in which hard ceramics based on titanium are uniformly and finely dispersed in the matrix on a base of high-speed steel. The rolling speed and life of the high-resistance alloy can be increased and lengthened sharply because this new alloy can be used in regions in which the cemented carbide has caused defects such as cracks, etc., due to the problem of toughness.

The main features of the new alloy are as follows: 1) toughness is extremely high even in regions of high hardness; 2) the new alloy is excellent in wear and welding deposition resistance because hard ceramics which cannot be manufactured by using a dissolving metallurgy method are uniformly dispersed in the matrix; and 3) the new alloy can even be formed in complex shapes because it is manufactured by the powder metallurgy method.

The new alloy is used to extrude aluminum, in thread rolling dies, and end mills, and as an alternative to cast iron rolling.

### Power Supply Unit for Carbon Dioxide Laser Oscillators in Full-Scale Production

Rofin Marubeni Laser Corp. is a joint concern established under joint management of Marubeni Corp., Nippei Toyama Research Institute, and West German Rofin-Sinar Laser GmbH which is one of the largest laser oscillator manufacturers in the world. The joint concern will start full-scale production of the power supply unit for carbon dioxide laser oscillators.

Marubeni Corp. has imported and sold carbon dioxide laser oscillators made by Rofin-Sinar Laser GmbH for 3 years, and has promoted the domestic production of the power supply unit for carbon dioxide laser oscillators since Rofin Marubeni Laser Corp. was established in August of last year.

Up to now, the joint concern has domestically produced three power supply units for oscillators with a laser output of 500 watts, andhas obtained data on characteristics equivalent to those of power supply units made in West Germany. Therefore, the joint concern, in April, will start mass producing three power supply units every month, and will supply them to Marubeni Corp. which is in charge of sales of power supply units. The joint concern will next set about the domestic production by July of the power supply unit for oscillators with a laser output of 1 kw and will start mass production in July.

The joint concern will import some parts such as vacuum pumps from foreign countries, but will use in the power supply unit main parts such as the body, PC boards, transformers, capacitors, and gas control units made in Japan. If the mass production of power supply units stays on track, the joint concern expects that the cost of power supply units compared to imported power supply units will be sharply decreased.

High-Performance Acoustic Copper Wire Which Achieves Sound Quality With Clarity

The Furukawa Electric Co., Ltd., has developed a high-performance acoustic copper wire, and has started manufacturing it. It is called "PCOCC (Perfect Crystal by Ono Continuous Casting) Material," and is made of high-purity oxygen-free copper which has little grain boundary. It is manufactured by using the OCC (Ono Continuous Casting) method developed by Atsumi Ono, a professor at Chiba Institute of Technology.

It is said that the sound quality with higher clarity can be obtained without any distortion of signals by using this PCOCC material in acoustic cables.

Up to now, copper wire materials have been manufactured by using a method in which molten copper is cooled and solidified in molds. But when this method is used, tensile force is caused by friction will act on the contact face between the mold and metal at the solidifying process, cracks will be formed on the surface of the metal materials, and metallographic defects will be caused by the air gap between the metal and the mold wall. For this reason, it had been necessary to scrape off fine defects from the surface to obtain high-quality products.

In contrast, when the PCOCC material is used, the molten metal and molding wall will come into contact with each other in the heated molds until just before the molten metal is solidified, and the molten metal acts as a lubricant. Therefore, after the molten metal is solidified, the surface of the PCOCC material will be an excellent mirror. Also, the PCOCC material will have a single crystal structure with a small amount of grain boundaries, because the internal structure has no formation of crystals from the molding wall and consists of a unidirectional solidified structure which grows from the center in the linear direction. Therefore, high-purity oxygen-free copper can be manufactured with a small amount of impurities.

## Superplastic Alloy Stretched 25 Times Its Length Will Be Commercialized in Two-Phase Stainless Steel

Sumitomo Metal Industries, Ltd., has developed a superplastic alloy which can be stretched to a maximum of 25 times its length at an extremely slow strain rate of $10^{-2}$ to $10^{-3}$ per second. This superplastic alloy is a two-phase stainless steel type of a new material based on iron, and has a mixed structure of ferrite and austenite.

Superplastic metal shows abnormally high ductility under specific conditions. Research on such superplastic metal is thriving in the field of nonferrous alloys such as aluminum and zinc alloys. Research on 1,000 percent superplasticity of an alloy consisting of three elements such as nickel, chromium, and iron has been reported in the field of ferrous alloys. It is expected that the two-phase stainless steel will be commercialized. The maximum elongation and strain rate of such two-phase stainless steel are 400 to 500 percent and are extremely small, being $10^{-4}$ to $10^{-5}$. Therefore, it had been regarded as difficult to use the superplasticity of steel materials.

The company has conducted research on superplasticity of two-phase stainless steel consisting of ferrite and austenite. After solid-solution-treating the two-phase stainless steel at a temperature of 1,250°C up to the present time, the company has developed the superplastic alloy cold-worked at 50 percent. It is said that this material separates out the ferritic phase during deformation at a temperature of 950°C and shows the high elongation of 2,500 percent or more. Also, it is expected that this new material will increase the range of new applications of iron materials used in chemical plants, and in parts of precision machines, because the new material possesses the higher joining efficiency for various metals, including carbon steel, than that obtained by welding.

## Mass Production of Stainless Ultrathin Bands With a Width of Up to 600 mm

Nisshin Steel Co., Ltd., will soon complete construction of a facility for mass producing stainless ultrathin bands at its Shunan Works in Yamaguchi Prefecture, and will begin sales in earnest.

This facility is a further developed version of the existing mill, and can mass produce ultrathin bands with a thickness of 100 microns and a width of up to 600 mm. The new facility is one of the four cold rolling mills (Sejimiya Mill) installed in the Shunan Works. Usually, general stainless cold-rolled steel plates are produced in the new facility, and when necessary, ultrathin coil lines will be installed to produce stainless ultrathin bands.

The company expects that these stainless ultrathin bands will be used to prevent noise in semiconductor circuits, and as magnetic shields.

## Mass Production of Ultrahigh Purity Niobium Which Has Attained "Five-Nines"

This year Toyo Soda Manufacturing Co., Ltd., will start mass producing the ultrahigh purity niobium which has attained the "Five-Nines" (99.9997 percent).

This niobium is used as a superconductive material in wiring materials for Josephson elements.

The usual and maximum purities of niobium put on the market at present are 99.5 and 99.99 percent, respectively. The method of manufacturing such niobium is as follows: iron oxide is added to pipe (chroa) ore and is reduced with aluminum. This is a method of mass producing niobium in which importance is attached to the cost. The melting point of aluminum is higher than that of niobium, and there is difficulty in the separation of tantalum, which has chemical characteristics similar to those of niobium.

After changing niobium with a market purity of 99.5 percent into a halide and refining the halide, the company has succeeded in separating tantalum by using a thermal CVD (chemical vapor deposition) method. As a result, when such niobium is used in wiring materials for LSIs, the tantalum, which is regarded as a factor in operational errors, contained in the niobium has been reduced from the conventional 700 to 2,000 ppm to less than 10 ppm. Also, it has become possible to separate completely from niobium impurities with high-melting points, such as molybdenum, tungsten, and vanadium.

At present, the company is producing the niobium at a test plant with a monthly production capacity of about 10 kg and is shipping samples. The company is planning to complete a factory with an annual production capacity of 100 kg of niobium within this year because users have evaluated the niobium highly.

There are two types of niobium products. One is a spherical product with a weight of 1 to 10 grams, and the other is a foil product with a thickness of 10 microns. The price for both types is ¥20,000 to 30,000 per gram.

High-Alloy Powder Made by Honganas AB is Sold for HIP.

Hoganas Gadelius Co., Ltd., has started selling high-alloy powder based on the GAPDRY (gas atomizer process dry method) developed by Hoganas AB in Sweden.

The GAPDRY is a method in which alloy powder is manufactured by spraying, drying, and cooling materials dissolved in a gas atmosphere nitrogen or argon. Hoganas AB has been manufacturing high-alloy powders such as Mcraly, Hecraly, Cocraly, and Nimonic 80A, by using this method. All the high-alloy powders are characterized by the fact that their oxygen, nitrogen, and sulfur content is extremely small compared with those made according to conventional methods.

Hoganas Corp. in the United States is a sister company of Hoganas AB. Including the affiliated company Hoganas AB has a share of more than 50 percent of the world market, and is the world's largest manufacturer of iron and steel powders. This largest company has completed a high-alloy powder manufacturing plant based on the GAPDRY method, and has established a system for supplying a wide range of high-alloy powders, to respond to need for high-quality alloy powders, and to increase the range of new applications of these alloy powders.

Hoganas Gadelius Co., Ltd., is a joint stock enterprise established by joint capitalization of Hoganas AB and Gadelius Co., Ltd., in April 1985. Its purpose is to increase the sales of products made by Hoganas AB in the Japanese market. This joint concern will increase the sales of products focused on plasma spraying high-alloy base metals and raw materials for HIP (hot isostatic pressing), because Hoganas AB has arranged a system for supplying the products.

## Ceramics

### Formation of Silicion Dioxide Film Achieved at Low Temperature by Using an Immersion-Deposition Method

Nippon Sheet Glass Co., Ltd., has developed a technology to form uniform silicon dioxide film with the hardness equivalent to glass. Such films can be formed by immersing objects containing special silicon compounds such as ceramics, metal, and plastics in an acid water solution.

The new coating technology indicates that when objects with various shapes are immersed in a solution with a low temperature (room temperature) up to about 40°C, silicon dioxide up to a maximum of 3 microns thickness can be deposited without cracking via an equilibrium reaction on the surface of these objects at a rate of about 550 angstroms per second. The deposited film is a high-purity film in which silicon and oxygen are arranged orderly, and has the hardness equivalent to glass even if it has not been subject to calcination.

Up to now, the CVD (chemical vapor deposition) method, the vacuum evaporation method, and the sputtering method have been used to form this type of film. To use these methods correctly, it had been necessary to form films at a high temperature, and there were many problems with the uniformity, density, and purity of the films and in the cost of equipment.

Use of the new coating technology has almost solved these problem points, and will make possible the formation of extremely smooth films without any defect on the surface of various objects with complex shapes. Also, it is anticipated that this new technology will be further developed mainly in the electronics industry, because the etching rate is low, being less than half that of the CVD method and the sputtering method.

### General-Purpose Carbon Fibers Are Manufactured by Using Coal Pitch as Raw Material

Osaka Gas Co., Ltd., in collaboration with Dainippon Ink & Chemicals, Inc., and Nippon Sheet Glass Co., Ltd., has developed a technology to manufacture general-purpose carbon fibers (short fibers) in which coal pitch is used as a raw material.

Up to now, a "centrifugal method" has been used to transform conventional pitch to short fibers. Now, instead of this "centrifugal method," a vortex method called "RGJ (rotary gas jet) method" has been introduced into the new

technology which has been commercialized. The RGJ method is a technology for manufacturing glass short fibers. It possesses the following features: 1) general-purpose carbon fibers can be manufactured continuously in the processes from fiberization to carbonization (commercialization of products), and the cost can be decreased by 20 to 30 percent as compared with the centrifugal method; 2) general-purpose carbon fibers with a thread diameter of 2 to 20 microns can be manufactured with the strength of 80 to 120 $kgs/mm^2$ and an elongation of more than 2.0; 3) the distribution of thread diameters is small, being less than 10 percent, while that of conventional products is about 15 percent; and 4) the quality is high.

The general-purpose carbon fiber is used mainly in electromagnetic shield materials, high-temperature heat insulating materials, and in reinforcements for cement, plastic, and rubber.

Osaka Gas Co., Ltd., will start operating a semicommercial plant with an annual production capacity of 10 tons, and will ship samples of general-purpose carbon fibers in the near future.

Full-Scale Production of Antibacterial Zeolite for Special Fibers

Shinagawa Fuel Co., Ltd., will produce antibacterial zeolite (alumina sodium silicate) excellent in deodorization on a full scale this summer.

This antibacterial zeolite had been patented by Kanebo, Ltd., and Yoshitsugu Hagiwara, an exprofessor of Tohoku University, but Shinagawa Fuel Co., Ltd., has obtained an exclusive license for this patent, has established a new company called "Shinanen New Ceramic Co., Ltd.," and has conducted R&D of the antibacterial zeolite with a view to putting it to practical use. Mr Hagiwara has been invited to join the new company.

Up to now, zeolite has frequently been used in Japan as a builder which dephosphorizes synthetic cleaners, but Shinagawa Fuel Co., Ltd., has developed the antibacterial zeolite by improving zeolite and giving it antibacterial and deodorizing properties. This antibacterial zeolite is impervious to bacteria and prevents the occurrence of mold because it is porous. Therefore, the company expects that the antibacterial zeolite kneaded in fibers can be used in the covers and sheets for bedquilts, white robes, wallpaper, and bathroom tile.

Plant Is Constructed To Synthesize Special Chemicals on the Basis of Technology for Using Zeolite

Toray Industries, Inc., has begun to build a specialty chemicals new synthesizing plant based on the use of zeolite technology at its Nagoya Plant. This new plant will be able to produce several kinds of articles such as methachlortoluene, 2,6-cyclotoluene and its derivatives, at a rate of 3,000 to 4,000 tons a year. It is a full-scale plant to be completed in July and start production in August.

These special substances are used in the fine chemical field as intermediate products of agricultural chemicals. Up to now, it has been necessary to remove further these substances, because it was impossible to obtain the amount of special substances which can be used industrially, and the special substances are mixtures, even if they are synthesized by using usual chemical methods.

In contrast, the company has succeeded in industrially producing special substances by isomerization with a zeolite catalyst followed by adsorption, separation, and removal. Up to now, the company has produced such substances at a pilot plant installed in the Nagoya Plant, but the company has decided to construct a full-scale plant using the opportunity presented by the decision to supply several European manufacturers of agricultural chemicals. Also, the company expects that the demand for special substances and the range of their applications will also be increased in Japan in the future.

CIP Efficiently Forms Powder Materials

Yuken Kogyo Co., Ltd., has developed a CIP (cold isostatic press) "YEP-2-100" which can efficiently press form such powder materials as ceramics.

The CIP is a unit designed so that high pressure is applied through liquid to the periphery of a rubber mold in which powder is packed as a raw material to form the powder in the specific shape. Also, the density of moldings will be uniform, because the liquid is formed in the specific shape by high pressure.

Conventional CIPs are devised so that a worker packs powder into a rubber mold and sets the rubber mold in a device, and after the rubber mold is pressure molded, the rubber mold is removed and the finished powder is taken out. It takes a worker about 20 seconds to set a rubber mold and to remove it, so the worker must monitor the CIP at all times.

The company has achieved the automation of all processes. That is, the company has flattened a cylindrical rubber mold of the conventional type, has incorporated two devices in the CIP, and has connected the interval between both devices and the pressurized sectionby using a conveyer. With regard to the two devices, one packs powder into a rubber mold, and the other takes out the finished powder from the rubber mold.

As a result, the time required from packing powder to taking it out of the finished powder has been reduced to 8.5 seconds which is less than half of the conventional time. In addition, the production efficiency will be enhanced and the cost of manufacturing ceramic products can be reduced sharply because it will be possible to carry out unmanned operation of the CIP at night.

Technology for Extracting Supercritical Gas Shortens Degreasing Process for Manufacturing Fine Ceramics

Sumitomo Heavy Industries, Ltd., has developed equipment which can shorten sharply the degreasing time by using a technology for extracting supercritical

gas by means of carbonic acid gas. Degreasing time has become a bottleneck in the process for manufacturing ceramic products. The degreasing time of a conventional heating system is 100 to 150 hours, but the new equipment requires only 2 to 3 hours.

When ceramics are injection molded to manufacture ceramic products, various kinds of resins, at 10 to 30 percent, are first added as binders to such ceramic raw materials as alumina, and silicon nitride, to simplify the injection molding process. After the mixture is formed, it passes through a degreasing process to take out the binder, and then is calcinated as the finishing process. However, considerable time is required to degrease the binder, and there are such problems as deformation and cracking of the molded product.

This new equipment can extract and degrease binder which is an organic material in a supercritical condition with the carbonic acid gas temperature of 45°C and a pressure of 200 kg/cm$^2$. It is said that 85 percent of the binder can be removed in 2.5 hours and there is no deformation in the products. Products with a weight of up to 20 kgs can be treated at one time. Also, the spent carbonic acid gas and binder can be recovered and reused.

Mass Production of Carbon Ceramics Which Can Be Machine-Cut

Nippon Tungsten Co., Ltd., has succeeded in mass producing carbon ceramics composed of carbon, silicon carbide, and boron carbide, and has started shipping samples.

The company will cultivate a market for such products by the brand name "CCE-1." These products are manufactured by industrializing composite carbon ceramics, which can be machine cut, and were developed by the Government Industrial Laboratory, Kyushu, of the Agency of Industrial Science and Technology of MITI.

The mass produced CCE-1 has a density of 1.9, the Shore hardness of 70, a deflection strength of 70 kg/mm, the electric resistivity of 5 milliohm-centimeters, and a thermal shock resistance of 600 $\Delta$T (degrees centigrade).

The CCE-1 usually can withstand a temperature of 1,000°C, and depending on service conditions, it is able to withstand a temperature of 1,200°C, and has excellent noncombustibility. Mechanical parts and complex products can be manufactured readily because they can be cut with a milling machine, and be finished with a grinding machine or lapping machine, to a condition close to that of a mirror surface, because of its high density. It is excellent in chemical resistance.

It is expected that the CCE-1 will be used in various fields such as mechanical parts, valves, electrical heating elements, electrodes, and biological materials.

C. Itoh & Co., Ltd., Obtains Selling Rights for Glass Ceramics Metallic Thick Plate Boards From Wade Advanced Ceramics Co., Ltd., in England

Wade Advanced Ceramics Co., Ltd. (England) has succeeded in industrializing glass ceramic metallic thick plate boards called "Keralloy" (brand name). C. Itoh & Co., Ltd., has obtained selling rights for the Keralloy in the Far East, the United States, and Oceania from the British company. Wade Advanced Ceramic Co., Ltd., is Britain's No 1 manufacturer of industrial ceramics used in alumina substrates, and valve balls.

The Keralloy is a compound obtained by coating special steel with ceramics based on glass. The special steel is an alloy of iron, chromium, and aluminum, developed by the Harwell National Laboratory in England, and has excellent bonding characteristics with ceramics. On the other hand, glass ceramics have been selected from the combination of lithium oxide, zinc, alumina, and silica, and the coefficient of thermal expansion matches that of the metal base.

There are several types of printed circuit boards. For example, epoxy is covered with copper foil, and conductive ink is printburned on alumina plates. Compared to these printed circuit boards, the Keralloy is excellent in heat radiation and does not need a heat sink which has been traditionally installed in electric parts to radiate heat from the electric parts. It is less expensive than the alumina board, because its price is determined by unit area, and the use of large alumina boards will double the price of usual alumina boards.

High Workability and High-Purity Silicon Nitride Is Manufactured at a Low Cost

Denki Kagaku Kogyo K.K. has developed a technology for refining and nitriding industrial metallic silicon with high purity at low cost, and has started shipping samples of this silicon to interested manufacturers.

This new technology is a method called "Unidirectional Solidification Refining." When a carbon crucible containing metallic silicon is heated while rotating in a high-frequency furnace, impurities such as aluminum, calcium, and iron are removed from the metallic silicon. The high-purity silicon nitride resistant to high temperature is manufactured by nitriding the high-purity silicon manufactured using this technology and by repeatedly pulverizing, refining, and drying this nitrided silicon.

This silicon nitride has been improved so that the calcium concentration, compared to conventional products, dropped from 2,400 ppm to 20 ppm, while that for aluminum dropped from 1,600 ppm to 160 ppm, and for iron, from 1,500 ppm to 50 ppm.

Up to now, a halogenation silicon nitriding method or a silica reducing method has been used to manufacture silicon nitride. When the halogenation method is used, silicon nitride will have high purity but will be expensive. On the other hand, when the latter method is used, silicon nitride will have a high-sintering characteristic, but will have a problem of low purity.

In contrast to these methods, when the new technology is used, silicon nitride will have such characteristics as high purity, high moldability, high sintering, and will be inexpensive. For this reason, it is anticipated that silicon nitride manufactured using this new technology will be expanded commercially for automobile engine parts such as turbochargers, cylinders, and connecting rods.

The development of the new technology is one of the articles included in the Research and Development Project of Basic Technologies for Next-Generation Industries of the AIST of MITI. This is the first time that the AIST of MITI has anticipated that one of the above articles will be put to practical use.

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NUCLEAR DEVELOPMENT

## SENSORS FOR NUCLEAR POWER GENERATION DISCUSSED

Tokyo SENSOR GIJUTSU in Japanese Aug 86 pp 18-21

[Article by Yoshiyuki Ara, Nuclear Reactor Measurement Laboratory, Japan Atomic Energy Research Institute]

[Text] The quantity of electricity generated by nuclear reactors currently accounts for 25 percent of the total electricity produced in Japan, and therefore plays a dominant role in government policies for the development of oil substitute energy and the diversification of energy resources which are regarded as vital in terms of national security. The disaster that occurred at the Chernobyl nuclear power station in late April, however, triggered once more the controversy over the safety of nuclear power stations. The cause of the accident, which is under investigation by both the Soviet authorities concerned and the International Atomic Energy Agency (Vienna), is expected to be released before long.

The nuclear power stations of Japan have been fortunate enough to have the least number of accidents in the world. They spend around 3 months on the obligatory periodic inspection and maintenance of the reactor, which is carried out annually by suspending operations. This is two to three times longer than those in the United States and European nations, and indicates that Japan exercises maximum efforts for ensuring safety which is based on the common concept of the people that no nuclear power generation is acceptable without it.

Meanwhile, sensors of various types, measuring systems, and signal processing systems corresponding to human sensory nerves, play an important role in ensuring the safety of nuclear power generation. The author outlines below the present status, objectives, features, future problems, etc., of sensor technology in connection with nuclear power generation.

### 1. Nuclear Reactor Instrumentation Sensors

The instrumentation for control of a nuclear power station is incorporated in the reactor itself, the primary and secondary coolant systems, the turbine, and in various accessory facilities. Most characteristic of these, however, is the instrumentation of the reactor which includes the primary coolant system. The sensors used in the reactor must be highly reliable and highly resistant to the environment unique to the reactor. Any objects inside and around a reactor are subjected to hazardous conditions such as high

temperature, high pressure, and high radiation, and sometimes to an intensely corrosive environment due to the presence of decelerators and coolants. Table 1 presents the environmental conditions of various types of nuclear reactors. All 33 nuclear power stations currently in operation in Japan are of the light-water reactor [LWR] type--16 units of BWR and 15 units of PWR--except for one GCR unit and one ATR unit. For FBR and VHTR, the nuclear reactors of the future, developmental research is underway.

Table 1. Conditions Dictating Nuclear Reactors

| Type of nuclear reactor | | Coolant (decelerator) | Temperature of the coolant at the outlet (°C) | Pressure of nuclear reactors ($kg/cm^2$) | Levels of radiation in the reactor | |
|---|---|---|---|---|---|---|
| | | | | | Neutron | γ-ray |
| Light water reactor | BWR | Light water | ≃300 | 70-75 | Thermal neutron flux $10^{12}$-$10^{14}$ $n/cm^2 \cdot s$ | $10^5$-$10^{10}$ R/h |
| | PWR | Light water | ≃320 | 157 | | |
| Fast breeder reactor (FBR) | | Liquid sodium | 500-650 | Normal pressure | | |
| Advanced thermal reactor (STR) | | Light water (heavy water) | 284 | 68 | | |
| Heavy water reactor (Canadian type) | | Heavy water (heavy water) | 290-300 | 80-110 | Fast neutron flux $10^{10}$-$10^{14}$ $n/cm^2 \cdot s$ | |
| Gas (cooled) reactor | | Carbon dioxide (graphite) | 330-410 | 8-20 | | |
| Advanced gas (cooled) reactor | | " " | ≃650 | ≃40 | | |
| High temperature gas (cooled) reactor | | Helium (graphite) | 750-850 | 10-50 | | |
| Multipurpose high-temperature gas cooled reactor | | " " | 900-1,000 | ≃40 | | |

The instrumentation of the nuclear reactor is generally comprised of three categories: 1) nuclear, 2) process, and 3) safeguard. Nuclear instrumentation is carried out with a view to monitoring the extent of the chain reaction of nuclear fission taking place inside the reactor, i.e., quantity of neutrons and γ-rays emitted as a result of the chain reaction. Various types of neutron detectors and γ-ray detectors, therefore, are available for this purpose. It is still necessary that the sensor introduced into the core of the reactor be of a small size; sensors with an external diameter of about 1-3 mm, which require no power source and are referred to as self-powered detectors, are practically used as are the ion-chamber type of sensors with external diameters of about 4-6 mm. These sensors can sufficiently resist

temperatures of over 300°C and an ion chamber resistant to temperatures of around 600°C–850°C has been developed in recent years.

Process instrumentation measures temperatures at various parts, pressures, water levels, flow volume of the coolant, among other things, within the reactor, thus playing an important role in its control. Process instrumentation sensors for LWRs do not differ significantly from those for industrial plants and fire power generation plants. However, requirements guaranteeing their quality are more stringent.

Finally, the safeguard instrumentation is a system for ensuring safety of the reactor and works independently of the other two instrumentations previously described. However, it has many measurement features similar to the nuclear and process instrumentations, and its sensors are made using identical technologies.

In any of the three instrumentations, instruments for measurements, including sensors, that are important for the operation and safety of nuclear reactors, have two- to fourfold systems of repetition (two out of three, two out of four, etc.). The following briefly reviews the FBR and VHTR, the reactors of the future, which are now under development. It is evident from Table 1 that these reactors are required to withstand more hazardous environments than the LWRs.

The FBR must withstand temperatures up to about 650°C and resist corrosion from liquid sodium. Here one finds unique measurement sensors of various types developed for liquid sodium; for example, the high electric conductivity of sodium is used in the large-aperture electromagnetic flow meter, small sodium thermometer-current meter of the eddy current type, sodium level gauge of the electromagnetic induction type, and quick response thermocouple which regards sodium liquids as one element wire of the thermocouple. Also developed are $LiNbO_3$ ultrasonic sensors for high temperature use which permits one to see through the opaque liquid sodium. Where the VHTR is concerned, development of a neutron detector operating at temperatures as high as 850°C–1,000°C is underway. A Pt-Mo alloy thermocouple, a thermal noise temperature sensor, and a stranded wire W-Re alloy thermocouple of the CA complex type are also under development.

## 2. Radiation Control Sensors

Work on the premises of nuclear power stations involving radioactivity inevitably exposes workers to the radiation which must be controlled so that the exposure stays below specified levels and the workers remain free of hazards. Also, the local inhabitants must be absolutely free from any exposure that could exceed permissible limits.

In order to achieve these objectives, radiation monitoring is comprised of three categories: 1) worksite monitoring, 2) personnel monitoring; and 3) environment radiation monitoring.

Worksite monitoring consists of continuous area monitoring and periodic monitoring using survey meters. Area monitoring is carried out for the purpose of monitoring the dosage rate in space and involves the measuring of $\gamma$-rays and leaking neutrons from the reactor. Periodic monitoring with survey meters, on the other hand, involves examining the degree of radioactive contamination in general in addition to that of the dosage rate in space and, therefore, takes measurements of $\alpha$-rays, $\beta$-rays, $\gamma$-X-rays, and neutrons. Radiation sensors incorporated in survey meters of various types are presented in Table 2.

Table 2. Major Radiation Sensors Used in Various Types of Survey Meters

| Classification | Sensor | Remarks |
|---|---|---|
| $\alpha$-ray use | ZnS (Ag) scintillator | Superior pulse-light selection with respect to $\beta$-ray |
| | Gas flow proportional counter (tube) | Small propane gas cylinder is incorporated. Among other gases available are methane and air |
| $\beta$-ray use | GM counter (tube) | Capable of distinguishing between $\beta$-ray and $\gamma$-ray |
| | Ion chamber | " " |
| | Scintillator | Stilbene, anthracene, plastics, etc. |
| X-ray use | GM counter (tube) | High sensitivity, available for quantitative determination |
| | Ion chamber | High energy correlation, available for quantity determination |
| | Scintillator | NaI (Tl), etc.; available for low dosage rate |
| Neutron use | $BF_3$ proportional counter (tube) | Available for thermal neutron; the decelerator paraffin is added for fast neutron |
| | Scintillator | For thermal neutron, LiI(Eu), plastics involving $^{10}B$ etc.; for fast neutron, plastics |

For personnel monitoring, various types of portable radiation sensors, as represented by filmbadges, pocket-size ion chamber dosimeters (TLD), etc., are used. Film badges are capable of separating $\beta$-rays or thermal neutrons from X-$\gamma$-rays, provided appropriate filters are used. The fluorescent glass dosimeter, which uses aluminophosphoric acid alkali glass, in turn, is highly sensitive to thermal neutrons because of the lithium and boron in the glass. The TLD, with its compact size, high sensitivity, and extended range of application, has recently found application in many of the nuclear power stations and is capable of separating thermal neutrons from $\gamma$-rays with cadmium plates

and $^{10}B$ films as filters. The major crystals involved in the TLD are LiF, $CaF_2$, and $CaSO_4$.

In environmental monitoring, meanwhile, monitoring posts or stations are set up around a nuclear power station at specified distances in order to measure the space dose rate outdoors and radiation involved in the dust in the air. The radiation sensor used for this purpose includes the Geiger-Mueller countertube, ion chambers, Nal(Tl) scintillation counters,and TLD.

3. Sensors for Judging and Investigating the Abnormality of the Reactor

The nuclear power station is required by law to suspend operations of the reactor once a year so that an in-service inspection (ISI) can be made. The ISI involves various nondestructive tests which consist largely of ultrasonic probing of damages in welded areas, etc., and eddy-current probing of damages in the heat transfer pipe of the steam generator. The eddy-current sensors and the ultrasonic sensors such as PZT, therefore, play important roles. The progress in damage probing technology is also remarkable, with the application of ultrasonic holography and the multiple frequency excitation method for eddy-current probing being tested.

The integrity of the reactor operation is also checked in the course of its operation. The loose part monitor, for example, is aimed at monitoring, by means of sound sensors and acceleration sensors, those sounds and vibrations produced by components of structures in the reactor which have become loose or fallen off. Sound sensors are used also for detecting abnormal boiling of the coolant and leakage from valves and pipes, whereas acceleration sensors are used for monitoring the vibration of rotating machines and equipment such as pumps and turbines.

It is expected that new technologies such as laser application sensors, infrared sensors, and other light sensors will see their applications rapidly expanding for such checking and examination of nuclear facilities.

4. Future Problems

Government policies to improve the technology of nuclear power generation by applying advanced technologies, including those of new raw materials, have been underway. One of them is the development and application of a robot that operates nuclear power reactors. Development of various types of sensors, resistant to radiation and incorporated in the robot, constitutes an important area of research, and upgrading the resistance of visual sensors against radiation is of particular importance. Nevertheless, CCD visual sensors, having been developed for that purpose and entering the stage of practical application, do not necessarily have adequate resistance against radiation. It is hoped that a superior CCD camera with sufficient resistance against radiation will be developed, not to mention its possible contribution to space development, with the cooperation of government and private organizations, though its market scale is far smaller than that for CCD cameras for general civilian applications.

Various amorphous materials, in turn, are gaining attention as raw materials and also as electronic elementary devices. Because of their amorphous or noncrystalline characteristics, these materials seem to be resistant to radiation. Development of new sensors resistant to radiation based on the characteristics of amorphous materials, therefore, makes a very intriguing area of research. It is believed that new sensor technologies are also being unveiled in the area of nuclear power.

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